

Anesthetic substances in the naphthalene series. V

Esters and alkylaminoalkylamides of 5-amino-1-naphthaleneacrylic acid. S. I. Sergievskaya and K. V. Levshina (All-Union Chem.-Pharm. Research Inst., Moscow). *Zhur Obshchel Khim.* (J. Gen. Chem.) 20, 1030-42 (1950); cf. C.A. 39, 11589. Gradual addn. of 10 g. $\text{Cu}(\text{Cl}_2\text{CH}_3)$ to 50 ml. HNO_3 (d. 1.47) at -5° to -0° and stirring 30 min. until room temp. was reached gave, upon filtration, 25% 5-nitro-1-naphthaldehyde (I), m. $130-7^\circ$ (from pyridine); diln. of the mother liquor with ice gave 17.8% 5-nitro isomer, m. $123-4^\circ$ (from EtOAc); yields of 20% and 22%, resp., are obtained when the entire mixt. is quenched with ice and the mixture recrystd. from pyridine. Heating 4 g. I, 4 g. malonic acid, and 10 ml. AcOH 12 hrs. at $85-90^\circ$ gave 3.9 g. (5-nitro-1-naphthylmethylene)malonic acid, m. $250-2^\circ$ (from 70% AcOH). Heating this 4 hrs. to 200° gave a 70% yield of 5-nitro-1-naphthaleneacrylic acid (II), m. $257-8^\circ$ (from EtOH), which is generally poorly sol.; it is also obtained by refluxing I and $\text{CH}_3\text{CO}_2\text{H}$, with pyridine, or by the Perkin reaction with AcO-NaOAc in 6 hrs. at $137-40^\circ$. II warmed with SOCl_2 yields the chloride (III), m. $132-3.5^\circ$ (from CaH_2). Stirring III with concd. NH_4OH at room temp. yields the amide, m. $227-8.5^\circ$ (from EtOH). II heated with MeOII in the presence of HgSO_4 8 hrs. gave the Me ester, m. $120-1^\circ$ (from MeOH); EtOH similarly gives the Et ester, m. $104-5^\circ$ (from EtOH). Treatment of II with KOH in EtOH , addn. of $\text{Cl}(\text{CH}_3)_2\text{Br}$, and refluxing 7 hrs. gave the 3-chloropropyl ester, m. $82.5-4.0^\circ$ (from EtOH). Heating III with cyclohexanol in CaH_2 3 hrs. gave the cyclohexyl ester, m. $85.5-6.5^\circ$ (from EtOH). Stirring the Et ester (1.3 g.) in 60 ml. EtOH at $57-60^\circ$ with 0.2 ml. concd. HCl and 1 g. Fe filings added over 1 hr., heating 6 hrs., and treating the crude product with

Ac_2O gave *EI* (β -acetamido-1-naphthaleneacrylate), $171.5-2.0^\circ$ (from EtOH). Refluxing 3 g. II 5 hrs. with 1.1 g. $\text{Me}_2\text{NCH}_2\text{CH}_2\text{OH}$ in CaH_2 gave the dimethylaminoethyl ester, m. 64° (from abs. EtOH/HCl salt, m. $198-9^\circ$ (from EtOH)); this reduced with $\text{Fe}-\text{HCl}$ in EtOH as above gave 2-dimethylaminoethyl 5-amino-1-naphthaleneacrylate-HCl, m. $194-1.5^\circ$ (from EtOH), which hydrolyzes on standing in d. aq. solns. The 2-diethylaminoethyl ester, m. $193-194^\circ$ (from EtOH), was prep'd. similarly from the intermediate *anato analog*, m. $199-201^\circ$ (from EtOH). Treatment of II with KOH in EtOH and heating the resulting K salt with $\text{Et}_2\text{N}(\text{CH}_2)_3\text{Cl}$ 3 hrs. at $80-5^\circ$ gave β -diethylaminopropyl 5-nitro-1-naphthaleneacrylate-HCl, m. $214.5-16^\circ$ (from 98% EtOH), reduced by $\text{Fe}-\text{HCl}$ in EtOH to the *anato analog*, m. $212-12.5^\circ$ (from EtOH). III in CaH_2 and $\text{Me}_2\text{NCH}_2\text{CH}_2\text{CH}_2\text{NH}_2$, warmed 4 hrs. on a steam bath gave the corresponding $N-(3\text{-dimethylamino-1-methylpropyl})$ amide, m. $111-5^\circ$ (from EtOH), converted by $\text{Fe}-\text{HCl}$ in EtOH to the *anato analog*, isolated as the *citrate monohydrate*, m. $126-8^\circ$ (from $\text{EtOH}-\text{Et}_2\text{O}$). Similarly, III gave the $N-(4\text{-diethylamino-1-methylbutyl})$ amide, m. $120-1^\circ$, converted to the corresponding 5-amino deriv., isolated as the *citrate monohydrate*, m. $133-5^\circ$ (from $\text{EtOH}-\text{Et}_2\text{O}$). Hydrogenation of $5\text{-}(4\text{-O}_2\text{NC}_2\text{H}_4\text{CH}_2\text{CHCO}_2\text{CH}_2\text{CH}_2\text{NEt}_2)_2\text{HCl}$ over Raney Ni in EtOH gave the corresponding ester of the 5-amino acid, isolated as the *HCl salt*, m. $150.5-60^\circ$ (from abs. EtOH). None of the amides gave complete anesthesia (rabbit cornea test), while the dimethylamino- and diethylaminoethyl esters of the 5-amino acid had weak anesthetic properties but caused tear formation and hyperemia; the diethylaminopropyl ester had some what better properties. G. M. K.

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Synthesis and transformations of 4-alkoxytetrahydro-1-naphthalenepropionic acids S. I. Sergievskaya and I. M. Lipovich (S. Ordzhonikidze Chem.-Pharm. Inst., Moscow), *Zhur. Obshch. Khim.*, 1, Gen. Chem.) 20, 1171 (1950); *J. Russ. Phys. Chem. Soc.* 43, 2195; 5,6,7,8-Tetrahydro-1-naphthol (I) (73 g.) in 125 ml. abs. EtOH contg. 31 g. KOH refluxed 3 hrs. with 177 g. MeI in 125 ml. abs. EtOH, yielded 62 g. *Me ether*, b_4 129 $^{\circ}$. Condensation of this with succinic anhydride gave *β*-*t*-methoxy-5,6,7,8-tetrahydro-1-naphthoylpropionic acid (II), m. 177 $^{\circ}$; this (5 g.) heated 6 hrs. with 50 ml. abs. MeOH and 1 ml. concd. H₂SO₄ gave 1 g. of the *Me ester*, m. 80-2.5 $^{\circ}$ (from EtOH-Me₂CO), hydrolyzed with alc. KOH to II, m. 177 $^{\circ}$. Addn. of 74 g. I to 31 g. KOH dissolved as much as possible in 125 ml. hot abs. EtOH, followed by addn. of 170 g. PrBr in 125 ml. abs. EtOH and refluxed 2-3 hrs., gave 70 g. *Pr ether*, b_4 111-2 $^{\circ}$; this (30 g.), 10 g. succinic anhydride, and 400 ml. dry PhNO₂, treated slowly with 18 g. AlCl₃ and let stand overnight, followed by stirring 6 hrs., gave on hydrolysis with ice water 32 g. crude 4-propoxy homolog of II, isolated via the Na salt and purified by heating the crude acid with MeOH in the presence of H₂SO₄, through the *Me ester*, m. 47-8 $^{\circ}$ (from EtOH), b_4 216-22 $^{\circ}$, which when heated 6 hrs. with EtOH-KOH gave the *free acid*, m. 134-7 $^{\circ}$ (from

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EtOH). Similar reactions yielded the *Bu ether* of I, b_4 168-70 $^{\circ}$, which condensed as above with succinic anhydride to give the crude 4-hexyloxy homolog of II, purified via the *Me ester*, b_4 228-32 $^{\circ}$, m. 35-6 $^{\circ}$ (from EtOH), which on boiling with alc. KOH yields the *free acid*, m. 114-17 $^{\circ}$ (from EtOH), the latter (5 g.) heated 21 hrs. with 30 ml. 1:1 HCl, 30 ml. AcOH, 30 ml. MePh, and 25 g. Zn-Hg (3 addn. of 1:1 HCl), 30 ml. AcOH, 30 ml. MePh, and 25 g. *tetrahydro-1-naphthalenebutyric acid*, m. 90-2 $^{\circ}$ (from 29 MeOH). A similar reaction sequence yielded the *heptyl ester* of I, b_4 178-80 $^{\circ}$, and the *Me ester*, b_4 215-26 $^{\circ}$, of the 4-heptyloxy homolog of II, m. 91-2 $^{\circ}$ (from EtOH); the same acid was obtained by adding 2.3 g. KOH in 30 ml. abs. EtOH to 10 g. *Et*-*β*-(4-hydroxy-5,6,7,8-tetrahydro-1-naphthoyl)propionate, heating 2 hrs. with 15 g. C₆H₅Br in EtOH (yield, 10 g. *Et ester*, b_4 215-18 $^{\circ}$), and saponif. the Et ester. The latter procedure with C₆H₅Br yielded the *Et ester*, b_4 235-40 $^{\circ}$, of the 4-hexyloxy homolog of II, m. 92-4 $^{\circ}$ (from EtOH). Heating 6 g. II 4 hrs. to 120-30 with 80 ml. AcOH and 20 ml. 48% HBr, followed by evap. *in vacuo* and extn. with alkali, gave 1 g. *4-HO acid*, m. 192-4.5 $^{\circ}$ (from C₆H₅Br), also obtained 11.5 g. (by heating 27 g. II in 1160 ml. C₆H₆ 5 hrs. with 55 g. AlCl₃ added slowly); the EtO and BuO acids give the same result. Refluxing the 4-HO acid with EtOH in the presence of H₂SO₄ gave the *Et ester*, m. 122-4 $^{\circ}$ (from MeOH). Reduction of the free acid by Clemmensen method gave 63% 4-hydroxy-5,6,7,8-tetrahydro-1-naphthalenebutyric acid, m. 127-30 $^{\circ}$ [from (CH₂Cl)₂].

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The synthesis and transformation of 4-alkoxycyclohexa-
1-naphthalenepropionic acids. S. I. Sergievskaya and I. M.
Lipovich. *J. Gen. Chem. U.S.S.R.* 20, 1915-22 (1950)
(Engl. translation).—See *C.A.* 45, 1590a. R. M. S.

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Methods of preparation of 1-naphthaleneacrylic acid and its ethyl ester. S. I. Sergeyevskaya, K. V. Levshina, and E. N. Petrova (S. Ordzhonikidze All-Union Chem.-Pharm. Inst., Moscow). *Zhur. Obshchel Khim.* (J. Gen. Chem.) 20, 1478-80(1950). A convenient synthesis of 1-C₆H₅CH=CHCOOH (I) is given as follows: Dried EtOAc (80 ml.) and 4 g. powd. Na cooled rapidly to 0° are treated with 1 ml. abs. EtOH, then with 20 g. 1-C₆H₅CHO at 0.5°, stirred 3 hrs., treated with 20 ml. AcOH, the ppt. dissolved by adding 60 ml. H₂O, and the soln. ext'd. with EtOAc, giving 21 g. (72%) 1-Et ester, λ_{max} 370.02 Å; this allowed to stand in abs. EtOH with 1.2 g. NaOH overnight gave the Na salt, which with HCl gave 8.9% of the free I, m. tenuis 203.5°, ϵ pure I, m. 208.10⁴ (from EtOH or MeOH). The yield of pure product is 43.7% (based on the aldehyde). If the Et ester is redistd. and allowed to stand, it solidifies and m. 37.8.5 (from EtOH), λ_{max} 390.7 Å; the yield of pure ester is 33% (based on the aldehyde). — G. M. K.

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5-Nitro-1-naphthalenepropionic acid and its transformations. S. I. Sergievskaya and K. V. Leyshina (S. Ordzhonikidze All-Union Chem.-Pharm. Inst., Moscow). *Zhur. Obshchey Khim.* (J. Gen. Chem.) **20**, 1481-6 (1950). Bromination of 2 g. $5,1-O_2NCH_2CH_2CH_2CO_2Et$ with 1.8 g. Br in $CHCl_3$ over 3 days gave 75% *disuboxide*, m. 147-8° (from EtOH). This (2.8 g.) in 75 ml. EtOH, treated at 35-40° with 2.8 g. KOH in 12 ml. EtOH and allowed to stand 12-15 hrs., gave a ppt. of *K, a-nitro-1-naphthalenepropionate*, which with dil. H_2SO_4 gave 0.65 g. *free acid* (4%, decomp. 182°) in a preheated bath (from $CHCl_3$), it sublimes partly at 155°; heating with EtOH and H_2SO_4 8 hrs. yields the *Ester*, m. 121-2° (from EtOH), which on reduction in EtOH-HCl with Fe and treatment with Ag_2O , yields *1,3-diaminod-1-naphthalenepropionate*, m. 183.5-4.0° (from EtOH). It heated with pyridine 1 hrs. to 100° yields *3-nitro-1-naphthylacetylene*, m. 156-7° (from EtOH). Warming I with excess $SOCl_2$ on a steam bath, removing the SO_2 by *vacuo*, and treating the residue with Et_3N - CH_2Cl -EtOH in $CHCl_3$ (1 hr.) gave 1,2-diethylenamethylder, isolated as the *HCl salt*, yellow solid, m. 180.2° (from abs. EtOH); reduction with Fe in EtOH-HCl at reflux temp. yielded the *3-amino analog*, isolated as the *citrate*, gray powder, fusing at 80°, liquefying at 90°, and forming a clear fluid at 135-40° (from abs. EtOH). The product was devoid of anesthetic values. G. M. K.

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The preparation of 1-naphthaleneacrylic acid and its ethyl ester. — S. I. Sergievskaya, K. V. Levshina, and E. N. Petrova. *J. Gen. Chem. U.S.S.R.* 20, 1539-41 (1950) (Engl. translation). — See *C.A.* 45, 2478d.
R. M. S.

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5-Nitro-1-naphthalenepropionic acid and its transformations
S. I. Serikovskaya and K. V. Levshina, *J. Gen. Chem. U.S.S.R.* 20, 1513-8(1950) (Engl. translation). See
R. M. S.

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Dealkylation of β -2-alkoxy-1-naphthoyl propionic acids and oxidation of β -2-ethoxy-1-naphthoyl propionic acid. S. I. Sergievskaya and A. A. Chemerinskaya (S. Ordzhonikidze All-Union Chem.-Pharm. Inst., Moscow). Zhur. obshch. Khim. (J. Gen. Chem.) 20, 2284 (1950). Heating 1 g. 2,1-MeOC₆H₄COCH₂CH₂CO₂H 5 hrs. with 2 g. AlCl₃ in 35 ml. C₆H₆ on a steam bath gave 0.7 g. β (2-ketoxy-1-naphthoyl)propionic acid (I), m. 116-17° (from H₂O); the EtO analog gave 0.6 g. Heating 2.5 g. 16 hrs. with 10 ml. EtOH and 1.2 ml. concd. H₂SO₄ gave 1.8 g. *Ester*, m. 92.3° (from EtOH). Clemmensen reduction of 0.3 g. I with 1.0 g. Zn-Hg, 1.5 ml. concd. HCl, 1.5 ml. AcOH, and 1.5 ml. MePh 20 hrs. (with 3 addns. of 0.5 ml. HCl) gave γ -hydroxy-1-naphthalenebutyric acid, m. 131.6° (cf. following abstr.). Heating 0.75 g. I-Ester 4 hrs. with 10 ml. 5% alc. KOH and 5 g. EtOH gave *Et* β (2-ethoxy-1-naphthoyl)propionate, m. 50.1° (from EtOH). 2,1-EtOC₆H₄CO₂H (7 g.) refluxed 1.5 hrs. with NaOCl (from Cl and 210 ml. 9% NaOH) gave 1.25 g. 2-ethoxy-1-naphthaleneglycolic acid (II), m. 159.61° (from EtOH), and NaOH-insol. matter from which 0.5 g. 2-ethoxy-1-naphthaldehyde (III), m. 109-10.5° (from EtOH), was isolated by extn. with Et₂O; the aldehyde boiled with PhNH₂ in EtOH 5 hrs. gave the *anil*, m. 71.3° (from EtOH). Heating II with EtOH in the presence of H₂SO₄ gave the *Ester*, m. 70.5-1.0° (from EtOH), giving the *free* II on hydrolysis with cold 6% alc. KOH. It yields a semicrystalline, m. 188.5, 0.5° (from MeOH), and 0.5 g. heated 1 hr. with 0.2 g. PhNH₂ in EtOH gave III, m. 108.10°. G. M. Kosolapoff.

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Synthesis, structure, and transformations of 3-(2-ethoxy-1-naphthoyl)propionic acids. S. I. Sergievskaya, A. V. Danilova, and A. A. Chemerinskaya (S. Ordzhonikidze All-Union Chem.-Pharm. Inst., Moscow). *Zhur. Obschch. Khim.* (J. Gen. Chem.) 20, 2314-20 (1950). Addn. of 120 g. AlCl₃ with stirring and cooling to 120 g. 2-C₆H₅CO₂H, 11. PhNO₂, and 63 g. succinic anhydride in 10 hrs., letting stand 8 hrs., and heating 3 hrs. to 40-5° gave, after the usual treatment of 67 g. mixed keto acids, m. 125-32°; esterification of this by refluxing 6 hrs. with 300 ml. EtOH and 30 ml. concd. H₂SO₄, and crystn. from EtOH gave 14.4 g. less sol. *EI* 3-(2-ethoxy-6-naphthoyl)propionate, m. 94-5°, and 11.2 g. *1-naphthoyl* analog, m. 51-2°. Hydrolysis by 0% alc. KOH gave the corresponding free acids, m. 102-3° (I), and m. 103-1° (II). If the Friedel-Crafts reaction is run in CS₂ the yield is much lower and the 1-C₆H₅CO isomer predominates. I purified by reesterification m. 104-5°; II, similarly purified, m. 101-5°. Oxime, m. 145-6°. I with NaOCl yields 2-ethoxy-6-naphthoic acid, m. 205-6°, which, heated with HBr-AcOH, gives the 2-HO analog, m. 240-1°

(acetate, m. 224-5°), yielding the *EI* ester (by refluxing in EtOH with H₂SO₄), m. 139-40-5° (from EtOH). Clemmensen reduction of the 2-HO acid over 20 hrs. gave 2-hydroxy-6-naphthalenebutyric acid, m. 154-7° (from aq. EtOH), also obtained by heating 2-methoxy-6-naphthalenebutyric acid, m. 131-5° (from the C₆H₅CO analog by Clemmensen reduction), 3 hrs. with AcOH-HBr. Clemmensen reduction of II gave 2-ethoxy-1-naphthalenebutyric acid, m. 93-4° (from aq. EtOH). Similar treatment of the 2-Me₂O analog gave 2-methoxy-1-naphthalenebutyric acid, m. 81-3°. Refluxing either acid with AcOH-HBr gave the 2-HO analog, m. 130.5-7.5° (from EtOH). Slow addn. of 20 g. 20% Na-Hg to 1.75 g. 2,1-MeOC₆H₄COCH₂CH₂CO₂H in 35 ml. 5% NaCO₃ over 8 hrs., stirring 2 hrs., sepn. of Hg, extn. of the oils with EtOH, acidification with 10% HCl, and boiling 10 min. gave 0.35 g. 3-(2-methoxy-1-naphthyl)butyric acid, m. 126-8° (from EtOH). The 6-naphthyl analog, prepd. similarly, m. 121-3°; 3-(2-ethoxy-1-naphthyl)butyric acid, m. 127-8° (from EtOH), and its 6-naphthyl analog m. 125-6°. G. M. Kosolapoff

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► The dealkylation of α -2-alkoxy 1-naphthoyl propionic acids and the oxidation of β -2-ethoxy 1-naphthoyl propionic acid. Yu. L. Svirskaya and A. A. Chumetskaya. *J. Russ. Chem. Soc.* 20, 2379 (1938) (Engl. transl., *See. C.* 1, 45, 7089).
B. L. M.

SURGIVSKII, I. I.

"Reduction of β -(alkoxyaryl) propionic acids and the preparation of (Alkoxyaryl) "butyrolactone." by I. M. Lipovich and S. I. Surgivskii. (p. 123)

SC: Journal of General Chemistry (Zhurnal Osnchsei Khimii) 19-1, Volume 1,
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α -2-Propoxy- α -butyloxy- δ -naphthoylpropionic acids and their transformations. S. I. Sergeyevskaya and A. A. Chernyskaya (S. Ordzhonikidze All-Union Chem. Pharm. Inst., Moscow), *Zhur. Obshch. Khim.* (USSR) Chem. ZN 30(1950) 1031. Addls. of 104 g. AlCl₃ with cooling to -10° g. 2-C₆H₅OOC-CH₂CH₂COOH, 0.8 g. succinic anhydride, and 1.1 L dry Ph₂O, stirring 5 hrs., and treatment with dil. HCl gave a 60% yield solid, m. 169-70°, while the filtrate yielded from the org. layer 26.5 g. *β -2-propoxy- δ -naphthoylpropionic acid* (I), m. 169-70.5° (from EtOH); heating with AcOH-HBr yields the 2-HO analog, m. 227-9°. Heating 1.6 hrs. with EtOH in the presence of H₂SO₄ gave the *Et ester*, m. 92-3.5° (from EtOH); the free E yields an *oxime*, m. 140.2° (from C₂H₅O). Clemmensen reduction (in MePh) gave after 20 hrs. about 30% *γ -2-propoxy-6-naphthalenebutyric acid*, m. 119.5-20.5° (from dil. EtOH); *Et ester*, m. 55.6° from EtOH. Slow addn. of 11 g. 20% Na-Hg to 0.9 g. I, add. 20 ml. 5% Na₂CO₃, and 10 ml. EtOH (this may be omitted with gradual addn. of 10% HCl, stirring 2 hrs., removal of the Hg, extn. with Et₂O, acidification of the aq. layer, boiling 10 min. extn. with Et₂O) and evapn., gave 0.35

g. *γ -2-propoxy-6-naphthyl- β -butyrolactone*, m. 127.5 (0.0 from EtOH). Heating 150 g. 2-C₆H₅OEt, 450 ml. BuOH, and 90 g. HgSO₄ 6 hrs. gave 88 g. 2-C₆H₅OEt, m. 31-32°. Dry 0.9 g. I and 28 g. succinic anhydride were gradually added to 88 g. AlCl₃ and 500 ml. PhNO₂, with ice cooling and stirred 100 hrs.; the usual treatment gave 8.4 g. *β -2-butyloxy- δ -naphthoylpropionic acid* (II), m. 150.2° (from EtOH), which with HBr-AcOH yields the 2-HO analog, m. 228-30°, while EtOH-H₂SO₄ treatment yields the *Et ester*, m. 82.3°, readily saponified to the original I, m. 150-1°, with alc. KOH; *Bu ester*, prep'd either by heating with BuOH in the presence of H₂SO₄, or by heating with BuHg in alc. KOH, m. 75.7° (tert.), m. 76.7-77° (from MeOH). II, *oxime*, m. 140.5° (decompn.) from EtOH. Clemmensen reduction of II (20 hrs.) yielded *γ -butyloxy- δ -naphthalenebutyric acid* (III), m. 114-16° (from EtOH), which, heated with AcOH-HBr yields the 2-HO acid, while heating with EtOH-H₂SO₄ yields the *Et ester* of III, m. 49-50° (from EtOH). Treatment of I as described above yields *γ -2-butyloxy- δ -naphthylpropionic acid*, m. 120-1° (from EtOH).

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Synthesis of the tetrahydronaphthalene series

Anilino ketones of the tetrahydronaphthalene series

1. 1-hydoro-4-(anisooecetyl)-5,6,7,8-tetrahydro-
naphthalene. S. I. Sergeeva-Yaya and R. G. Vitolina (All-
Union Chem. Pharm. Inst., Moscow). Zhur. Obshch.
Khim. i. Gen. Chem. 1, 21, 888 (1951). — 1-Hydoro-
acetyl-5,6,7,8-tetrahydro-naphthalene (10 g.) in 50 ml. *Ke-*

l-decanone, m. 115-10° (from EtOH); this (10 g.) in 50 ml. *CHCl₃* was brominated with 1.8 ml. Br, warmed to 25-30° briefly, then evap'd. in vacuo, and heated to remove the excess Br and HBr, yielding 12.57 g. 1-hydoro-4-(bromoacetyl)-5,6,7,8-tetrahydro-naphthalene (11), m. 124-9° (decomp.).

From *CHCl₃*. The structure is proved by condensation with *CH₃COCl* (*N*-sa derivative) and hydrolysis with 10% aq. aq. KOH, yielding (5,6,7-ketotetrahydro-4-hydroxy-1-methyl-2-methylenoic acid), m. 157-0°, which heated in pyridine to 120-5° gave authentic *β*-3,5,7-trimethoxy-4-hydroxy-1-naphthylpropanoic acid, m. 160-1°. (*E* ester, m. 121-3°, 1 (9 g.) in 27 ml. *CaH* stirred 3 hrs. with 0.8 g. PhCO₂Na/H₂O, then kept stand overnight, gave 95% *β*-CH₃CO₂NHMe₂HBr, m. 165-0°, while the filtrate on evapn. and trituration with abs. Et₂O, then treatment with dry HCl, gave 32% 1-methoxy-4-(bromomethyl)naphthalene-5,6,7,8-tetrahydro-*β*-naphthyl-*β*-HCl, m. 192-4° (from *MeCO-NaOEt*). Purple, yellow, m. 137-8° (from EtOH). Refluxing 10-10 hrs. with 30% *MeOH*-aq. HCl gave 50% free 1-HO analog, decomps. 198-201° (from EtOH), while the 1*z* derivative, with H over *Pd-C* in *MeOH*, gave 50% 1-hydoro-4-methylenoic acid, 5,6,7,8-tetrahydro-4-naphthalene-*β*-HCl (II), decomps. 221-3° (from EtOH). Similar hydrolysis of the 1HO derivative, gave 10.5% 1-hydoro-4-(methylenoic acid)-5,6,7,8-tetrahydro-naphthalene-*β*-HCl (III), decomps. 202-3.5° (from EtOH), also formed from the 1*z* derivative by refluxing 10-12 hrs. with 30% aq. *MeOH*-HCl. I (3 g.) in 20 ml. dry *CaH*, mixed with 0.5 g. *MeCNH*, 3 hours gave 1-hydoro-4-methyl-5,6,7,8-tetrahydro-naphthalene as a salt, m. 201-5° identical with II. Refluxing this with 30% aq. *EtOH*-HCl (anisooecetyl)-5,6,7,8-tetrahydro-naphthalene-*β*-HCl, m. 202.5-4.0° (from *MeCO-NaOH*), whose *aq* soln. above (rapid hydrolysis; similar hydrolysis of the 1-HO analog gave 10% 1-hydoro-4-(anisooecetyl)-5,6,7,8-tetrahydro-naphthalene-*β*-HCl, decomps. 205-7° (from EtOH-Et₂O). Condensation of I with Et₃NH in *CaH* in 4.5 hrs. gave 80% crude, 42% pure, 1-hydoro-4-dimethylnitroso-5,6,7,8-tetrahydro-*β*-naphthyl-naphthalene-*β*-HCl, decomps. 181.5-3.5° (from ab. EtOH), whose *aq* soln. readily hydrolyzed; purple, m. 122-3° (from EtOH-Et₂O); hydrolysis with 30% *MeOH*-HCl for 12 hrs. gave 60% 1-hydoro-4-(dimethylnitroso)-5,6,7,8-tetra-

β-naphthyl-naphthalene-*β*-HCl, decomps. 203-5° (from *MeOH*).

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Amino ketones of the tetrahydronaphthalene series.
Synthesis of 1-hydroxy-4-(aminoacetyl)-5,6,7,8-tetrahydro-naphthalenes. S. I. Sergievskaya and R. G. Vllovina. *J. Gen. Chem. U.S.S.R.* 21, 675-83 (1951) (Engl. translation). See *C.A.* 46, 9574. B. R.

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"(N-Alkyl-N-Hydroxyethyl)-Amides of 4-(5)-Nitro-1-Naphthoic Acids," S. I. Sergiyevskaya, Ye. N. Petrova, All-Union Sci Res Chemicophar Inst imeni S. Ordzhonikidze

"Zhur Obshch Khim" Vol XXI, No 12, pp 2174-2178.

Prepd (N-butyl-N-hydroxyethyl)- and (N-isobutyl-N-hydroxyethyl)-amides of 4-nitro-1-naphthoic acid and (N-isobutyl-N-hydroxyethyl)-amide of 5-nitro-1-naphthoic acid. Carried out amide-ester rearrangement with certain of these compds.

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SERGIYEVSKAYA, S. I.

USSR/Chemistry - Pharmaceuticals

Feb 52

"ac-Tetrahydronaphthoic and ac-Tetrahydrothionaphthoic Acids and Their Derivatives," S. I. Sergiyevskaya, N. P. Volynskiy, All-Union Sci Res Chem-Phar Inst imeni S. Ordzhonikidze, Moscow

"Zhur Obshch Khim" Vol XXII, No 2, pp 321-328

Prepd simplest derivs of ac-tetrahydronaphthoic acids (not described in the literature) and their alkylaminoalkyl esters. Found that mp of amide of ac- α -tetrahydronaphthoic acid is 168°, not 116°C as indicated in the literature. Prepd ac-tetrahydrothionaphthoic acids, their ethyl and alkylaminoalkyl esters.

209T30

C A
Organic Chemistry 10

Anesthetic substances of the naphthalene series. VII.
Monalkylaminonaphthalene esters of some naphthalene carboxylic acids. S. I. Sergievskaya and E. N. Petrova (S. Ordzhonikidze Chem. Pharm. Inst., Moscow). *Zhur. Obshch. Khim.* (J. Gen. Chem.) **22**, 328-33 (1952); cf. *C.A.* **45**, 2458; **46**, 2003. Heating 3.25 g. 4,1-O₂NCH₂CH₂COCl 24 hrs. at 55° with 1.5 g. PrNHCH₂CH₂OH in CHCl₃ satd. with HCl gave 2-propylaminonaphthalene-4-nitro-1-naphthoate-HCl m. 173-4° (from EtOH); reduction over Raney Ni in MeOH gave the 4-amino analog, m. 208-8.5° (from EtOH). Similarly were obtained the following 2-alkylaminonaphthalene-4-nitro-1-naphthoate-HCl salts and the p-amino analogs (m.ps. in brackets): iso-Pr, m. 175-6° (200.5-1.0° (from EtOH)); Bu, m. 162.5-3.0° (from EtOH) [206-7° (from EtOH)]; iso-Bu, m. 152-3° (from EtOH) [m. 181-2° (from EtOH)]; heptyl, m. 148-9° [m. 191-2° (from EtOH)]. 5,1-O₂NCH₂CH₂COCl with BuNHCH₂CH₂OH in CHCl₃ satd. with HCl gave 2-butylaminonaphthalene-5-nitro-1-naphthoate-HCl, m. 197-8°, reduced to the 5-amino compd., m. 217-20°. Similarly was obtained 2-isobutylaminonaphthalene-5-nitro-1-naphthoate-HCl, m. 220-1°, which could not be reduced satisfactorily. Heating 1-C₆H₅CH₂CH₂COCl with BuNHCH₂CH₂OH in CHCl₃ satd. with HCl gave 2-butylaminonaphthalene-1-naphthaleneacrylate-HCl, m. 139.5-40.5°; iso-BuNHCH₂CH₂OH gave the iso-BuNHCH₂CH₂ ester-HCl, m. 182-3° (from EtOH). The products were prep'd. for evaluation as anesthetics.

G. M. Kosolapoff

USSR/Chemistry - Pharmaceuticals

Mar 52

"Synthesis of m-Amino (Hydroxy)-Phenyl- β -(Methylamino)-Ethanols and Catalytic Reduction of m-Nitroacetophenone," S. I. Sergiyevskaya, G. A. Ravel', All-Union Sci Res Chem-Phar Inst imeni S. Ordzhonikidze

"Zhur Obshch Khim" Vol XXII, No 3, pp 496-502

m-Aminophenyl- β -(methylamino)-Ethanol was obtained. Its structure was shown by its transformation into the hydrochloride of the known m-hydroxyphenyl- β -(methylamino)-ethanol. Gives a

209T48

USSR/Chemistry - Pharmaceuticals (Contd)

Mar 52

method for obtaining m-hydroxyphenyl- β -(methylamino)-ethanol, and describes a method for obtaining m-amino-acetophenone by a catalytic conversion.

209T48

USSR/Chemistry - Synthetic Drugs

Jun 52

"Nitration of ac-1-Tetrahydronaphthoic Acid and Transformations of Nitro-ac-1-Tetrahydronaphthoic Acids," S. I. Sergiyevskaya, N. P. Volynskiy, All-Union Sci Res Chem-Phar Inst imeni Ordzhonikidze, Moscow

"Zhur Obshch Khim" Vol XXII, No 6, pp 1035-1047

In nitration of ac-1-tetrahydronaphthoic acid with HNO₃ of sp gr 1.5, 5,7-dinitro-(1,2,3,4-tetrahydro)-1-naphthoic acid is formed; in nitration with HNO₃ of sp gr 1.4, a mixt of 2 mononitroacids, 5- and

218r26

USSR/Chemistry - Synthetic Drugs (Contd) Jun 52

7-(1,2,3,4-tetrahydro)-1-naphthoic acids, is obtained. A method for sepg them is given. Their structure was clarified by their conversion into compds with known structures. By means of catalytic reduction, the corresponding diamino- and amino-ac-1-tetrahydronaphthoic acids were obtained from them. The simplest derivs of all nitro(amino)-ac-1-tetrahydronaphthoic acids were prep'd. 5(7)-Nitro-ac-1-tetrahydronaphthoic acids and their derivs were obtained.

218r26

SERGIYEVSKAYA, S. I.

SERGIEVSKAYA, S.I.; VOLYNSKIY, N.P.

Nitration of 1,2,3,4-tetrahydro-2-naphthoic acid. Zhur. Obshchey
Khim. 22, 1446-50 '52.
(CA 47 no.13:6387 '53)

I. S. Ordzhonikidze All-Union Chem.Pharm. Inst., Moscow.

SERGEIEVSKII, S. I.

Chemical Abst.
Vol. 48 No. 5
Mar. 10, 1954
Organic Chemistry

Nitration of 1,2,3,4-tetrahydro-2-naphthoic acid. S. I.
Sergeevskii and N. P. Volynskii. *J. Gen. Chem. U.S.S.R.*
22, 1489-91(1952)(Engl. translation).—See C.A. 47, 6388a.
H.L.H.

chem ②4

32T29

USSR/Chemistry - Pharmaceuticals

Sep 52

"Dehydrogenation and Oxidation of 2-Ethoxy-(5,6,7,8-tetrahydronaphthoyl-3)-propionic Acid,"
S. I. Serfiyevskaya, I. M. Lipovich, All-Union
Sci Res Inst imeni S. Ordzhonikidze, Moscow

"Zhur Obshch Khim" Vol 22, No 9, pp 1650-1655

The constitution of 2-ethoxy-(5,6,7,8-tetrahydronaphthoyl-3)-propionic acid was detd more precisely by oxidizing this compd into 2-ethoxy-3-tetrahydronaphthoic acid and subsequent conversion of the latter into compds of known

232T29

structure. The dehydrogenation of 2-ethoxy-(5,6,7,8)-tetrahydronaphthyl-3-butyric acid was carried out in the presence of palladium deposited on carbon.

232T29

Chem Abs v47

1-25-54

Organic chemistry

Synthesis of 1,1-phenylcyclopentanecarboxylic acid and 1,1-phenylcyclopentanenitrocarboxylic acid and some of their derivatives. K. V. Levshina and S. I. Serfiyevskaya (S. Ordzhonikidze All-Union Chem. Pharm. Inst., Moscow). "Zhur. Obshch. Khim." 22, 2189-93 (1952). To 1530 ml. 48% HBr is carefully added 307 ml. concd. H₂SO₄ and the mixt. treated gradually with 172 g. tetrahydrofuran after which the mixt. is heated on an oil bath gradually up to 100-5° at which it is kept 20-3 hrs. (temp. in the mixt.); the lower layer and CHCl₃ ext. of the top layer yield 70% (BrCH₂-CH₂), b₁, 85-90° (I). To 87 g. NaNH₂ in 1.3 l. dry MePh is gradually added 109.3 g. PhCH₂CN at below 35°, the mixt. is stirred 5-10 min. and 200 g. I is added at 50-5°, after which the mixt. is stirred at 90-2° 24 hrs., cooled, treated with H₂O to take up ppt'd. NaBr and the org. layer and MePh ext. of the aq. layer are combined and distd., yielding 110 g. (70%) 1,1-phenylcyclopentanecarbonitrile (II), b₁, 145-60°. II (80 g.) and 450 ml. 20% KOH in MeOH heated in rotating autoclave 6 hrs. at 195-200° (35-40 atm. pressure), cooled, concd., dild. with H₂O, extd. with (CH₂Cl)₂, and filtered and acidified with 250 ml. 1:1 HCl, gave 95% crude 1,1-phenylcyclopentanecarboxylic acid, m. 157-8° (from EtOH), m. 159°; Na salt, needles, m. above 220°; acyl chloride (III), b₁, 149-50°. III (44.7 g.) in 350 ml. C₂H₆ added gradually to KSH soln., obtained by satn. of 25.3 g. KOH in 14 ml. H₂O and 447 ml. EtOH with H₂S

at -5° stirred 20-30 min. filtered, and concd., yielded 65-70% K 1,1-phenylcyclopentanethiocarboxylate, m. 166-7° (from EtOAc), which with 10% HCl yields 85% free acid (IV), m. 43-4° (from EtOH). III (11 g.) in C₂H₆ at -5° with soln. of 5.5 g. KOH in 100 ml. EtOH and 3 ml. H₂O satd. with H₂S (5-6 g. wt. gain), filtered and the ppt. washed with H₂O, gave RCOSCOR, C₁₁H₁₂O₂S, m. 143-4° (from EtOAc). IV (3 g.) in EtOH acidified to Congo red with HCl, and treated with alc. FeCl₃ gave 1.5 g. RCOSSCOR, C₁₁H₁₂O₂S, m. 82-3.5° (from EtOAc-EtOH). Heating K salt of IV with EtI in abs. EtOH gave the Et ester, b₁, 134°; similarly Et₂NCH₂CH₂Cl gave the diethylaminomethyl ester; HCl salt, m. 137-7.5° (from EtOAc). Et₂N(CH₂)₂Cl gave the diethylaminopropyl ester; HCl salt, m. 132-3° (from EtOAc-EtOH). G. M. Kosolapoff

11A
L-23-54

Chemical Abst.
Vol. 48 No. 9
May 10, 1954
Organic Chemistry

② Chen

The synthesis of 1-phenylcyclopentanecarboxylic acid and 1-phenylcyclopentanediocarboxylic acid and some of their derivatives. K. V. Lezhina and S. I. Serevskaya. J. Gen. Chem. U.S.S.R. 22, 2247-50 (1952) (Engl. translation).—See C.A. 46, 584g.

H. L. H.

SERGIEVSKAYA, S.I.; VOLYNSKIY, N.P.

2-Naphthoic acid from 2-iodonaphthalene. Zhur. Priklad. Khim. 25, 898-9
'52. (MLRA 5:8)
(CA 47 no.20:10514 '53)

1. S.Ordzhonikidze All-Union Chem.-Pharm. Inst., Moscow.

СЕРГИЕВСКАЯ, С. И.

Chemical Abst.
Vol. 48 No. 6
Mar. 25, 1954
Organic Chemistry

Condensation of *ar*-1-methoxytetrahydronaphthalene with chlorides of fatty acids. S. I. Sergievskaia and A. A. Kropacheva (S. Ordzhonikidze All-Union Chem. Univer-

tit, Moscow). Zhur. Obshchel. Khim. 23, 463-6 (1953); cf. C.A. 40, 7186. — *ar*-1-Methoxytetrahydronaphthalene (I) (30 g.) in 240 ml. PhNO₂ treated with 11.5 g. AcCl, then at 0° with 30 g. AlCl₃, stirred 4 hrs., allowed to stand 24 hrs. at room temp., and treated with ice-HCl gave 82.6% Ms. *ar*-1-methoxytetrahydro-4-naphthyl ketone, b₁ 147-8°, b₂ 152-3°. This (5 g.) refluxed 6 hrs. in dry C₆H₆ with 10 g. AlCl₃ and then treated with ice-HCl gave 3.8 g. *HO* analog, m. 154-5°. Similarly 80 g. I with 60 g. EtCOCl and 75 g. AlCl₃ gave 26 g. Et *ar*-1-methoxytetrahydro-4-naphthyl ketone, b₁ 169-71°, m. 36-7°; with AlCl₃ in C₆H₆ as above yielded 55% *I*-HO analog, m. 152-3°; attempt to cleave the MeO link by refluxing the ether in AcOH with 48% HBr gave only a low yield of tetrahydro-*ar*-1-naphthol, m. 63-5°. Similarly I and PrCOCl gave 67.9% Pr *ar*-1-methoxytetrahydro-4-naphthyl ketone, b₁ 181-8°, converted as above to the *I*-HO analog, 60.7%, m. 131-2°. I add palmitoyl chloride similarly gave pentadecyl *ar*-1-methoxytetrahydro-4-naphthyl ketone, b₂ 214-16°, an oil which solidified on standing. G. M. Kosolapoff

SERGIYEVSKAYA, S.I.

1-Nitration of 1-propynaphthalene and 4-nitro-1-propynaphthalene; 4-amino-1-propynaphthalene and some of its derivatives. S. I. Sergeevskaya and G. Ya. Uretskaya (S. Ordzhonikidze All-Union Sci. Research Clinico-Pharm. Inst., Moscow). Zhur. Obshchey Khim. 23, 1542-54 (1953). RMgBr from 207% g. 1-C₆H₅Br treated with 121 g. CH₃-CH₂Br in 300 ml. C₆H₆ gave 60% 1-C₆H₅CH₂CH=CH₂. b.p. 134-5°, which hydrogenated over Raney Ni or EtOH gave 63% 1-C₆H₅Pr, b.p. 134.5°, d₄²⁰ 1.004(?) n_D²⁰ 1.5928. This (30.9 g.) treated during 1 hr. with 62 ml. HNO₃ (d. 1.4) at 40°, then stirred 20-30 min., and the 80% yield of crude material repeatedly distd., gave 14.5 g. 4-nitro deriv., b.p. 143-4°, m. 34-5°. Along with a liquid residue of higher boiling nitro deriv. Hydrogenation of the 4-nitro deriv. in EtOAc over Raney Ni gave 80% 4-amino deriv., b.p. 174-5°, b.p. 154.5°. Ac deriv. m. 133°; Bz deriv., m. 185-6°; N-Et₂C deriv., m. 69.5-70°; N-isopropyl-C deriv., m. 105.5-6.5°; N-MeC₃C deriv., m. 67-8°. Treatment of the amine with p-AcNHCO₂H₅SO₃Cl in pyridine at 55-60° gave 1,4-Pr₂C₆H₄NHSO₃H₅Cl-p, m. 182.5-3.0°, which, heated 2 hrs. with 13% NaOII yielded 1,4-Pr₂C₆H₄NHSO₃H₅Cl-p, m. 175.5-6.5°.

G. M. Kosinlapoll

$$\sum_{i=1}^n \lambda_i^2 < +\infty \Leftrightarrow \sum_{i=1}^n i^{-2} < +\infty$$

APPROVED FOR RELEASE: 08/23/2000

CIA-RDP86-00513R001548120016-3"

Catalytic method of preparation of 1-methylnaphthalene from 1-chloromethylnaphthalene. S. I. Selegnyeva, G. Ya. Uretskaya, and T. S. Safonova (S. Ordzhonikidze All-Union Sci. Research Chem.-Pharm. Inst., Moscow). *Zhur. Obshchey Khim.*, 23, 1027-30 (1953).—Hydrogenation of 1- $\text{ClCH}_2\text{C}_6\text{H}_4$ (I) over Raney Ni in the presence of alc. NaOH yields 60-70% 1-Me C_6H_4 , 1-C $\text{H}_5\text{N}_H\text{CH}_2\text{OEt}$ (II) and bis(1-naphthylmethane) (III). With dry NaOAc in EtOH the reaction yields 55% 1-Me C_6H_4 . Shaking 17.3 g. I in 200 ml. alc. NaOH and 10 g. Raney Ni in H atm. gave 59% 1-Me C_6H_4 , b.p. 310-13° (picrate, m. 141-1.5°), and 0.3 g. higher boiling materials. I (50 g.) in 14.5 g. NaOH and 350 ml. EtOH, shaken with 10 g. Raney Ni at 27.5 atm. H₂ at room temp. gave 20.9% 1-Me C_6H_4 , and about 7 g. II, b.p. 130-5°. The high boiling fractions from both expts. gave III, m. 164°. Pure II, b.p. 133-3°, b.p. 134-3°. I (10 g.), 7.3 g. dry NaOAc and 100 ml. 95% EtOH shaken with 10 g. Raney Ni in H atm. at atm. pressure gave 78.7% 1-Me C_6H_4 and a little III. The same reaction run at 25 atm. H₂ gave in 1 hr. 85% 1-Me C_6H_4 , and small amounts of II and III. I (10 g.), 15 ml. EtOAc, 3.7 g. NaOH and 100 ml. 95% EtOH shaken with 10 g. Raney Ni at atm. pressure gave 86.2% 1-Me C_6H_4 , and 1 g. III. The reaction run at 25 atm. H₂ gave 85% 1-Me C_6H_4 and a little III.

PERSHIN, G.N., laureat Stalinskoy premii, professor, redaktor;
SHCHUKINA, M.N., professor, redaktor; NATRADZE, A.G., otvetstvennyy
sekretary'; SERGIYEVSKAYA, S.I., professor, chlen redaktsionnoy
kollegii; MAGIDSON, O.Yu., professor, laureat Stalinskoy premii,
chlen redaktsionnoy kollegii; UTKIN, L.M., professor, chlen redaktsion-
noy kollegii; MASHKOVSKIY, M.D., professor, chlen redaktsionnoy kolle-
gii; KARAKHANYAN, O.I., redaktor; GLUKHOYEDOVA, G.A., tekhnicheskiy
redaktor.

[Synthomycin] Sintomitsin. Otvet. red. G.N.Pershin. Moskva, Gos.
izd-vo med. lit-rv. 1954. 194 p. (MLRA 7:8)

1. Moscow. Nauchno-issledovatel'skiy khimiko-farmatsevticheskiy
institut.
(Chloromycetin)

SERGIYEVSKAYA, S.I.

[Chemistry and medicine; anesthetics of the naphtalene and tetro-hydronaphthalene series] Khimiia i meditsina; anestesiiruiushchie veshchestva riada neftalina i tetragidronaftalina. Moskva, Medgiz, 1954. 197 p.
(Anesthetics) (Naphthalene)

(MLRA 8:2)

SERGIEVSKAYA, S. I.

SERGIEVSKAYA, S. I.

USSR/Chemistry

Card 1/1

Authors : Leveshina, K. V.; and Sergievskaya, S. I.

Title : Derivation of aliphatic-aromatic N-bis-(ethyl chloride)-amines

Periodical : Zhur. Ob. Khim. 24, Ed. 5, 905 - 909, May 1954

Abstract : The derivation of new aliphatic-aromatic compounds, namely benzyl-N-bis-(ethyl chloride)-amines with alkoxyl groups in the aromatic nucleus is described. Certain other analogous compounds were also synthesized. The most suitable method in the synthesis of benzyl-N-bis-(ethyl chloride)-amines is the one in which aromatic chloromethylated compounds ($ArCH_2Cl$) were the basic substances. The success of the synthesis depends to a greater extent upon the availability of the basic chloromethylated compound and its properties. New data are presented on the methods of obtaining certain basic substances. Six references (all English and German). Tables.

Institution : The S. Ordzhonikidze All-Union Scientific-Research Chemical-Pharmaceutical Institute, Moscow, USSR

Submitted : December 3, 1953

SERGIYEVSKAYA, S. I.

✓ Nitration of *br-5,6,7,8-tetrahydronaphthalene-2-carboxylic acid* and the transformations of 1-nitro- and 4-nitro-5,6,7,8-tetrahydronaphthalene-2-carboxylic acids. S. I. Sergiyevskaya and E. G. Popova (S. Ordzhonikidze All-Union Sci. Research Chem. Pharm. Inst., Moscow). *Zhur. Obshchel Khim.* 25, 2154-61 (1955). — To 43 ml. HNO₃ (d. 1.6) was added with stirring and cooling over 45 min. 35.2 g. *br-tetrahydronaphthalene-2-carboxylic acid* at 0°; after 30 min. the mixt. was filtered, yielding 31.1% *1-nitro-5,6,7,8-tetrahydronaphthalene-2-carboxylic acid* (I), m. 221° (from CCl₄-CH₂Cl). The mother liquors from purification of this gave the crude *4-nitro analog*; this was esterified with EtOH-HCl and the crude product, treated with EtOH, left behind the less sol. *Et I ester*, m. 107-8°; the soln. was distd. yielding the *Et ester* of the *4-nitro analog*, b. 180-6°; hydrolysis with 0.5N KOH gave 5.4% *4-nitro-5,6,7,8-tetrahydronaphthalene-2-carboxylic acid* (II), m. 200° (from MeOH). If the nitration of the carboxylic acid (15 g.) is done in 65 ml. concd. H₂SO₄ with 8.7 g. KNO₃ and 20 ml. H₂SO₄ at 0° there is formed 5 g. I and 0.7 g. II. Hydrogenation of over Ni in EtOH gave 78% *1-amino-5,6,7,8-tetrahydronaphthalene-2-carboxylic acid* (III), decomp. 185°; II similarly gave *4-amino analog* (IV), m. 192-3. These on heating

(2)

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with Ba(OH)₂ are readily decarboxylated; treatment of the amines with Ac₂O gave, resp., 1-acetamido-5,6,7,8-tetrahydronaphthalene, m. 158°, and the 4-acetamido analog, m. un-
stated. III diazotized in aq. HCl and heated with much H₂O to 40-50° gave 1-hydroxy-5,6,7,8-tetrahydronaphthalene-
2-carboxylic acid, m. 166.5-7.5°; IV gave the 4-hydroxy
analog, m. 102-4°. I refluxed with EtOH-HCl gave 85%
Et ester, b₁ 164°, m. 109°; MeOH gave the Me ester, m.
149-50°. I with SOCl₂ gave the acyl chloride, m. 97-8°;
amide, m. 218-19.5°; hydrazide, m. 163°. II gave: Et ester,
m. 82-2.5°; Me ester, m. 59.5-60°; acid chloride, m. 60-1°;
b₁ 161-2°, hydrazide, m. 181.5-2°. Hydrogenation of I Et
ester over Raney Ni at 20 atm. gave III Et ester (HCl salt, m.
105-7°), which with Ac₂O gave Et 1-acetamido-5,6,7,8-tetra-
hydronaphthalene-2-carboxylate, m. 122.5-3.5°. I hydrazide
hydrogenated over Raney Ni to III hydrazide, m. 147-8°.
II Et ester hydrogenated thus to IV Et ester, m. 65°. II
hydrazide gave IV hydrazide, m. 138.5-9.5°; IV with Ac₂O
gave 4-acetamido-5,6,7,8-tetrahydronaphthalene-2-carboxylic
acid, decomp. 293°.

G. M. Koslapoff

SER'-IYEVSKAYA, S.I.

Some transformations of 1-amino-5,6,7,8-tetrahydro-
naphthalene-2-carboxylic acid. S. I. Sergievskaya and
E. G. Popova. *J. Gen. Chem. U.S.S.R.* 25, 2203-4 (1955)
(Engl. translation).—See C.A. 50, 9427h. R. M. R.

SERGIYEVSKAYA, S.I.

✓ Some transformations of 1-amino-5,6,7,8-tetrahydro-naphthalene-2-carboxylic acid. S. I. Sergievskaya and E. G. Popova (S. Ordzhonikidze All-Union Chem. Pharm. Research Inst., Moscow). *Zhur. Obshchel Khimi.* 25, 2240-2 (1955); cf. *C.A.* 50, 8504d. Heating 15 g. 1-amino-5,6,7,8-tetrahydronaphthalene-2-carboxylic acid with 30 ml. AcO 20 min. at 80° gave 91% 2-methyl-7,8,9,10-tetrahydro-*III*-naphthal[1,2-d]-*n*-oxacin-1-one, m. 122-4° (from EtOH). This (12 g.) kept 2 days with a slight excess of 5% NaOH, filtered, and acidified to Congo red gave 12.9 g. 1-acetamido-5,6,7,8-tetrahydronaphthalene-2-carboxylic acid, m. 183-4° (decompn.) (from $\text{C}_2\text{H}_5\text{CH}_2\text{Cl}$). The same forms from the amino acid and AcCl in pyridine; the same product is obtained from the amino acid on heating with AcOH 3.5 hrs. at 100°. C. M. Kosolapoff

SERGIEVSKAYA, S. I.

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Alkylaminoalkyl esters of 1-amino- and 4-amino-5,6,7,8-tetrahydronaphthalene-1-carboxylic acids. S. I. Sergievskaya and E. G. Popova. J. Gen. Chem. U.S.S.R. 25, 2379-81 (1955) (English translation). See C.A. 50, 6355h.

B. M. R.

RM

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SERGIYEVSKAYA, S.I.

Alkylaminoalkyl esters of 1-amino- and 4-amino-5,6,7,8-tetrahydronaphthalene-2-carboxylic acids. S. I. Sergievskaya and E. G. Popova (S. Ordzhonikidze All-Union Chem. Plurin. Sci. Research Inst., Moscow). *Zhur. Obshchel Khim.*, 25, 2488-92(1955); cf. *C.A.*, 39, 701¹; preceding abstr.—Reaction of the acyl chlorides with *ω*-diethylaminoalkanols in C_6H_6 gave the following esters: 2-diethylaminoethyl 1-nitro-5,6,7,8-tetrahydronaphthalene-2-carboxylate-HCl, m. 172-3°; Me_2N analog-HCl, m. 167-8°; 2-diethylaminoethyl 4-nitro-5,6,7,8-tetrahydronaphthalene-2-carboxylate-HCl, m. 170-2°; Me_2N analog-HCl, m. 181-2°. Heating the free acid with $R_2N(C_2H_5)_2Cl$ on iso-PrOH gave: 39.6% 3-diethylaminopropyl 1-nitro-5,6,7,8-tetrahydronaphthalene-2-carboxylate-HCl, m. 161-3° (reduced with H over Raney Ni to the 1-amino analog-HCl, m. 186-7°). Similar hydrogenation gave: 2-diethylaminoethyl 1-amino-5,6,7,8-tetrahydronaphthalene-2-carboxylate-HCl, m. 150-7°; 2-diethylaminoethyl 4-amino-5,6,7,8-tetrahydronaphthalene-2-carboxylate-HCl, m. 164-8°; the Me_2N analog-HCl, m. 195-6°; 3-diethylaminopropyl 1-amino-5,6,7,8-tetrahydronaphthalene-2-carboxylate-HCl, m. 124° (decomp.). *Et* 4-amino-5,6,7,8-tetrahydronaphthalene-2-carboxylate treated in C_6H_6 with $Et_2N\cdot CH_2CH_2Cl$ 5 hrs. at reflux gave *Et* 4-(2-diethylaminoethylamino)-5,6,7,8-tetrahydronaphthalene-2-carboxylate-HCl, m. 162-4°. G. M. Kosolapoff

USSR

Some derivatives of 4-amino-1-methylnaphthalene. S.
I. Sergievskaya and G. Yu. Uretskaya. *J. Appl. Chem.*
U.S.S.R., 28, 109-11 (1955) (Engl. translation).—See *C.A.*
49, 7637b. H. L. H.

Card 1/1

Pub.

Authors : Sergiyevskaya, S. I. and Uretskaya,

Title : Some derivatives of 1-naphthalenemethylamine

Periodical: **APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548120016-3"**
no. 1, 115-118, 1955

Abstract : A method of nitration of 1-methylnaphthalene, which yields 30% of 4-nitro-1-methylnaphthalene is given. Preparation of some derivatives of naphthalenemethylamine is described. Two references (1 Russian: 1947)

Institution: All-Union Chemopharmaceutical Scientific Research Institute (im. S. Ordzhonikidze) in Moscow

Submitted : My 30, 1953

SERGIEVSKAYA, S. I.

Chem ✓ Derivatives of 4-hydroxy- and 4-butoxy-5,6,7,8-tetrahydronaphthalene-2-carboxylic acids. S. I. Sergievskaya and E. G. Popova (S. Ordzhonikidze All-Union Chem.-Pharm. Inst., Moscow). *Zhur. Osnovnoi Khim.* 26, 155-6; 1958.

Gen. Chem. U.S.S.R. 26, 155-7(1958)(Engl. translation).—
4-Hydroxy-5,6,7,8-tetrahydronaphthalene-2-carboxylic acid (I) (12 g.) in 120 ml. MeOH was treated with 6 ml. H₂SO₄, followed by dry HCl (10% wt. increase) and refluxed, 12 hrs, yielding 96% *t*-Me ester, m. 127.5-8.5°. I heated with Ac₂O gave the *t*-acetate, m. 180-1°. I Me ester refluxed in MeEtCO with Bu₁ and powd. K₂CO₃, 10 hrs. gave 70% *t*-butoxy-5,6,7,8-tetrahydronaphthalene-2-carboxylate, m. 49-50°, which was sepd. from residual starting material by its greater solv. in petr. ether. Hydrolysis of this with alc. KOH gave 82% *t*-Bu ether of I, m. 166-7°. This with SOCl₂ gave 84% acyl chloride, b.p. 180-2°, m. 47.5-8.5°, which with the appropriate amino alc. in hot C₆H₆ gave the 2-diethylaminoethyl ester, m. 148.5-9.5°, and 2-dimethylaminoethyl ester, m. 125-6°. I Et ester, m. 110-11°; hydrazide, m. 211-13°; iso-Am ester, m. 57-9°, b.p. 206-8°.

G. M. Kosolapoff

~~SERGIEVSKAYA, S.~~
SERGIEVSKAYA, S. I.

Alkaroxylic amino alcohols. I. Synthesis of α -(*m*-amino-*o*-phenyl)- β -alkylaminoethanols. S. I. Sergievskaia and L. E. Sventitskaya (S. Ordzonikidze All-Union Chem. Pharm. Research Inst., Moscow). *Zhur. Obshchei Khim.*, 26, 1967-76 (1950). — To 30.5 g. *m*-O₂NCH₂COCH₂Br suspended in 150 ml. C₆H₆ was added at 15–18° 30.25 g. PhCH₂NHMe in 30 ml. C₆H₆ and after 2 hrs, 200 ml. Et₂O was added and the pptd. PhCH₂NHMe·HBr was filtered off. The filtrate treated to Congo red with Et₂O-HCl gave 94.5% *m*-O₂NCH₂COCH₂NMeCH₂Ph·HCl (I), decomp. 173–4°. Similarly was obtained: 5% *m*-O₂NCH₂CO-CH₂NEt₂CH₂Ph·HCl, decomp. 154–4.5° (from EtOH); 93.9% α -O₂NCH₂COCH₂N(CH₂Ph)CHMe₂·HCl, decomp. 155.5–6.5°; 76.7% *m*-O₂NCH₂COCH₂N(CH₂Ph)Pr·HCl, decomp. 164.5°; 44.4% *m*-O₂NCH₂COCH₂N(CH₂Ph)Bu₂·HCl, decomp. 115–18°; 78.2% *m*-O₂NCH₂COCH₂NAm-CH₂Ph·HCl, decomp. 138.5–9.5°. Hydrogenation of I in MeOH over Raney Ni at 35 atm. H and 76° 2 hrs. gave PhCH₂NHMe and about 30% liquid, b₆₅ 115–55°, which on fractional pptn. from Et₂O gave 2 products: *m*-H₂NC₆H₄CH(OH)CH₂NHMe (II), m. 84–5°, and *m*-H₂NC₆H₄CH(OH)Me, m. 68–9°. If the hydrogenation was run over Pd in EtOH contg. 5% H₂O and 1.4% concd. HCl, there was obtained 82.8% II, m. 84–5°; *N,N'*-diacetyl deriv. (III), m. 152.5–3.5°. This amino alc. acidified with Et₂O-HCl to Congo red and the resulting oil taken up in EtOH, pptd. with Me₂CO, and shaken with excess KOH gave 74.2% 2,2,3-trimethyl-5-(*m*-amino-*o*-phenyl)tetrahydronazole (crude, b₆₅ 127–40°), m. 95.5–6.5°, which with Ac₂O readily gave III, while heating the oxazole deriv. with EtOH-HCl followed by treatment with NH₃ yielded 75.1% II. Hydrogenation of

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Sergievskaya, S. I., . . .

the remaining nitro derivs. over Pd as above gave *m*-H₂N-C₆H₄CH(OH)CH₂NRCH₂Pk (R shown): 88.8% *Ei*, m. 80-1° (*di-Ac* deriv., m. 133-3.5°); 53.8% *iso-Pr*, m. 80.5-80-1° (*di-Ac* deriv., m. 130-8.5°); 50% *Pr*, m. 74-5° (*di-HCl salt*, decomp. 203-4°); 82.4% *Bu*, m. 79.5-80° (*di-HCl salt*, decomp. 212-13°); 74.2% *Am*, m. 87-8° (*di-HCl salt*, decomp. 227-8°). II. α -(*p*-Aminophenyl)- β -alkyl-aminoethanols. *Ibid.* 2077-81.—Addn. of 24.2 g. PhCH₂NHMe in 25 ml. C₆H₆ at 15-18° to 24.4 g. *p*-O₂NC₆H₄COCH₂NMeCH₂Ph.HCl, decompr. 178.5°, similarly were prep'd.: *Ei*, 32.8%, m. 145.5-6.5°; *iso-Pr*, 33%, m. 150°; *Pr*, 14.3%, m. 102.6°; *Bu*, 23%, decomp. 146.5-47°; *Am*, 31.6%, decomp. 140.6°. Hydrogenation in EtOH over Pd gave β -H₂N-C₆H₄CH(OH)CH₂NH₂ (R given): *Me*, m. 124-4.5°; *Ei*, m. 134.5-5.5°; *iso-Pr*, m. 137-8°; *Pr*, m. 89.5-90°; *Bu*, m. 80-90°; *Am*, m. 72.5-73°. The *Me* deriv. gave the *di-HCl salt*, decomp. 120°; the *Bu* deriv. yielded the *di-HCl salt*, m. 138-7°. The *di-HCl* salts of the remaining amino alcs. were analyzed but the m.p.s. were not given. III. Methods of preparation of α -(*m*-hydroxyphenyl)- β -(methylamino)ethanol. S. I. Sergievskaya, A. A. Kropacheva, and L. E. Sventitskaya. *Ibid.* 2332-5.—Hydrogenation of 20 g. *m*-HO-C₆H₄COCH₂NMeCH₂Ph.HCl in 145 ml. MeOH with 0.3-0.4 g. Raney Ni at 50-60° under 20-30 atm. H₂ gave, after sepn. on the Ni, evapg. the MeOH, decolorizing in H₂O with active C, cooling, and adding 10 ml. 20% NH₄OH, *m*-HO-C₆H₄CH(OH)CH₂NHMe, which with HCl gave the *HCl salt* (I), 70%, m. 142-5° (from EtOEt-Me₂CO), *m*-H₂N-C₆H₄CH(OH)CH₂NHMe (3.5 g.) in 25 ml. 10%

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HCl treated with 0.5N NaNO₃ at 5° (or below), heated 30 min. to 75°, concd. *in vacuo*, taken up in EtOH, filtered, evapd., taken up in H₂O, and treated with NH₃ gave 70% I, m. 142-4°. Shaking 12.8 g. *m*-O₂NC₆H₄-COCH₃NMeCH₂Ph.HCl in 75 ml. H₂O with 1.28 g. PdCl₄ at 35° in H with 5 ml. H₂O and 2.3 ml. concd. HCl gave after 4.5 hrs. reaction, followed by sepn. of Pd, addn. of HCl and NaNO₃ (finally heating to 75°), 49% I, m. 142-4° free base, m. 167-8°. (G. M. Kosolapoff)

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SERGITEVSKAYA, S.I. ; SVENTSITSKAYA , L.Ye.

Research in the field of aliphatic-aromatic amino alcohols. Part2.
 α - (*p*-aminophenyl)- β -alkylaminoethanols. Zhur. ob. khim. 26
no.7:2077-2081 Jl '56. (MIRA 9:10)

1. Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy
institut imeni S. Ordzhonikidze.
(Ethanol)

SERGIYEVSKAYA, S.I.; KROPACHEVA, A.A.; SVENTSITSKAYA, L.Ye.

Study of fatty-aromatic amino alcohols. Part 2: Methods for the preparation of α -(*m*-hydroxyphenyl)- β -methylaminoethanol. Zhur. ob. khim. 26 no.8:2322-2325 Ag '56. (MLRA 10:11)

1. Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy institut imeni S. Ordzhonikidze.
(Ethanol)

See file: 1548120016-3

Alkylaromatic amino alcohols. III. Methods of preparation of α -(*m*-hydroxyphenyl)- β -(methylamino)ethanol. S. I. Serebryanskaya, A. A. Kropacheva, and L. E. Sventsitskaya. *J. Gen. Chem. U.S.S.R.* 26, 2597-9 (1956) (English translation).—See *C.A.* 51, 5047e. B. M. R.

*See file
1548120016-3*

SERGIYEVSKAYA, S. I.

Chem

Alkylated diamides of the stilbene series. I. M. Lipovitch and S. I. Sergievskaya (S. Ordzonikidze All-Union Chem. Pharm. Research Inst., Moscow). *Zhur. Obshchey Khim.* 26, 3402-4 (1956).—Addn. with cooling of an EtOH soln. of the appropriate amine to the di-HCl salt of the 4,4'-di-(imino ether) of the appropriate stilbene gave after 1 day in the cold a ppt. which after clarification with C in hot H₂O gave the desired diamide as the di-HCl salt on acidification with 10% HCl and cooling. Thus were prep'd. 64% 4,4'-di(methylamidino)-2HCl, m. above 340°; 50% 4,4'-di(methylamidino)-2-hydroxystilbene-2HCl, m. above 320°; 43% 4,4'-di(butylamidino)-2-hydroxystilbene-2HCl, decomp. 300-11°; 31% 4,4'-di(diethylamidino)-2-hydroxystilbene-2HCl, m. 300-2°; 50% 4,4'-diamidino-2-nitrostilbene-2HCl, m. above 320°; 47% 4,4'-dimethylamidino-2-nitrostilbene-2HCl, decomp. 320°; 50% 4,4'-diamidino-2-aminostilbene-3HCl, decomp. 315-18°; 10% 4,4'-di(methylamidino)-2-chlorostilbene-2HCl, m. above 320°; 50% 4,4'-di(methylamidino)-2-iodostilbene-2HCl, m. above 320°; 35% 4,4'-di(benzylamidino)-2-hydroxystilbene-2HCl, decomp. 314-10°. It was found that in the Sandmeyer conversion of p,p'-diaminostilbene to the dicyano anil, the diazonium soln. is best added to the Cu₂(CN)₃ soln. with cooling and in the presence of C₆H₆ to eliminate the otherwise bothersome foaming.

C. M. Kosolapoff

SERGIYEVSKAYA, S. I.

Chen

Preparation of 1-alkylnaphthalenes. S. I. Sergievskaya and T. S. Safonova (S. Ordzhonikidze All-Union Chem. Ind. Res. Inst., Moscow), Khim. 26, 3479-85 (1956). — A satisfactory method for 1-alkylnaphthalenes was developed on the basis of dehydration of corresponding alkamols by hydrogenation over Ni. Dehydration of butanol in KOH led to isomerization of group. Distn. of 1-C_nH_nCHMeOH (38.5 g, KHSO₄) and 0.25 g. hydroquinone on open fl. product, b₁ 93-0°, identified as largely 1-vinylnaphthalene, b₁ 93-4°, picrate, m. 101°. This hydrogenated over Raney Ni in EtOH at room temp. and atm. pressure gave 76% 1-ethylnaphthalene, b₁ 109-10°, d₄ 1.02 (picrate, m. 99°). Similarly 1-C_nH_nCH₂OH gave the olefinic product, b₁ 98-100° (picrate, m. 110°), which on hydrogenation gave 78.0% 1-PrC₆H₅, b₁ 0.9902, n_D 1.6001; picrate, m. 93°. Similarly 1-C_nH_nCMeOH gave 27.5 g. olefinic product, b₁ 103-6°, n_D 1.5872 (picrate, m. 89-90°), which gave 1-PrC₆H₅, b₁ 120-1°, picrate, m. 88°. 1-C_nH_nCH₂Cl, b₁ 51.8° (RB) with 20.0 g. PrCHO gave 4.5% CHPrOH, b₁ 190-1° (picrate, m. 51-2°), which is above to the olefin, b₁ 143-5° (picrate, m. 91-2°), which gave 95.8% 1-C_nH_nBu, b₁ 148.5-0°, d₄ 0.97 (picrate, m. 94-5°). Oxidation of the olefin gave 1-C_nH_nCO₂H confirming the structure. distd. from powd. KOH at 30 mm.; there is b₁ 130-40°, which does not contain 1-(a-naphthyl)butanol. On hydrogenation yields 66.8% pure 1-*n*-butylnaphthalene, b₁ 148.5-0°, d₄ 0.97 (picrate, m. 207-8°), from 1-C_nH_nCH₂Cl, b₁ 51.8° (RB).

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75-95% yield
on the basis
in KHSO₄ and
1-naphthyl-1-
the aliphatic
) from 0.2 g.
me gave 27 g.
1-vinylnaphthalene,
ad over Raney
Ni in EtOH at room temp. and atm. pressure
15 min. gave
8. n_D 1.6069;
gave the olefin
(which on
hydrogenation gave 78.0% 1-PrC₆H₅, b₁
0.9902, n_D 1.6001; picrate, m. 93°. Similarly 1-C_nH_nCMeOH gave 27.5 g. olefinic product, b₁ 103-6°, n_D 1.5872 (picrate, m. 89-90°), which gave 1-PrC₆H₅, b₁ 120-1°, picrate, m. 88°. 1-C_nH_nCH₂Cl, b₁ 51.8° (RB) with 20.0 g. PrCHO gave 4.5% CHPrOH, b₁ 190-1° (picrate, m. 51-2°), which is above to the olefin, b₁ 143-5° (picrate, m. 91-2°), which gave 95.8% 1-C_nH_nBu, b₁ 148.5-0°, d₄ 0.97 (picrate, m. 94-5°). Oxidation of the olefin gave 1-C_nH_nCO₂H confirming the structure. If the alc. is
distd. from powd. KOH at 30 mm.; there is b₁ 130-40°, which does not contain 1-(a-naphthyl)butanol. On hydrogenation yields 66.8% pure 1-*n*-butylnaphthalene, b₁ 148.5-0°, d₄ 0.97 (picrate, m. 207-8°), from 1-C_nH_nCH₂Cl, b₁ 51.8° (RB).

G. M. Kosolapoff

79-2-32/53

AUTHORS: Sergiyevskaya, S. I. and Safonova, T. S.

TITLE: About the Nitration of 1-n-Butylnaphthalin (O nitrovenii 1-n-butilnafatalina)

PERIODICAL: Zhurnal Obshchey Khimii, 1957, vol 27, No 2, pp. 421-428 (U.S.S.R.)

ABSTRACT: The reaction of nitration of 1-n-butylnaphthalin, 4-nitro-1-n-butyl-naphthalin and 4,5-dinitro-1-n-butylnaphthalin in nitric acid of various concentration was investigated under different conditions. Separation of the nitro compounds formed as a result of nitration was accomplished by means of the chromatographic adsorption method. Temperature and nitric acid concentration had a positive effect on the formation of mono-, di- and trinitro compounds during the nitration of 1-butylnaphthalin. During the nitration of 4,5-dinitro-1-butylnaphthalin, the authors encountered conditions in which trinitro-1-butylnaphthalin was the sole reaction product. Consequent conversion of 1-butylnaphthalin through 4-nitro-1-butylnaphthalin into 4,5-dinitro-1-butylnaphthalin and 4,5-dinitro-1-butylnaphthalin into trinitro-1-butylnaphthalin showed that the two nitro groups in the latter compound are in 4 and 5 positions of the naphthalin ring. The position of the third nitro group in trinitrobutylnaphthalin is explained.

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AUTHORS: Sergiyevskaya, S. I. and Safonova, T. S. 79-2-33/58

TITLE: 4-Amino-1-n-Butylnaphthalin and its Conversions (4-Amino-1-n-butylnaphthalin i yego prevrashcheniya)

PERIODICAL: Zhurnal Obshchey Khimii, 1957, vol 27, No 2, pp. 428-431 (U.S.S.R.)

ABSTRACT: 4-Amino-n-butylnaphthalin was obtained by catalytic reduction of 4-nitro-1-n-butylnaphthalin in the presence of a skeletal nickel catalyst. The catalytic reduction also yielded the hydrochloride, acetyl and benzoyl derivatives of this compound. The derivation of 4-chloro-1-n-butylnaphthalin, 4-iodo-1-n-butylnaphthalin and 4-n-butyl-1-naphthoic acid is described. The 4-amino-1-n-butylnaphthalin is described as a liquid substance, rapidly darkening when exposed to air, particularly when heated. Distillation of this substance was possible only in a nitrogen atmosphere in the presence of antioxidants. 4-n-butyl-1-naphthoic acid was obtained from 4-iodo-1-n-butylnaphthalin by means of the Grignard reaction.

Card 1/2 1 Reference, which is Slavic.

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Aromatic amino alcohols. IV. Pharmacologically active aryltetrahydrooxazoles. S. I. Sergiyevskaya, L. E. Sventitskaya, and Yu. I. Sviridov. *Zh. Org. Khim.* 1971, 7, 1511-1514.

Reaction of 2,3-dimethyl-3-phenyl-5-(*m*-aminobenzyl)tetrahydrooxazole, m.p. 168°C, with $\text{NH}_2\text{NH}-\text{H}_2\text{N}$ (1 g) in MeOH (20 ml). Heating with $\text{NH}_2\text{NH}-\text{H}_2\text{N}$ followed by cooling, followed by filtration and evapn gave 1.5 g 2,3-dimethyl-3-phenyl-5-(*n*-aminobenzyl)tetrahydrooxazole, m.p. 168°C in EtOH . Heating 1 g $\text{MeNHCH}_2\text{CH}(\text{C}_6\text{H}_5\text{OH}-m)-\text{OH HCl}$ with 1 g BzH_2 , a little K_2CO_3 , and excess MeOH 2 hrs. similarly gave 1.2 g 2-phenyl-3-methyl-5-(*m*-hydroxybenzyl)tetrahydrooxazole, m.p. 168°C (HCl salt, colorless crystals). The products showed pharmacological activity.

V Nitration of 1-styrylnaphthalene, 4-nitro, and 1,3-dinitro.

27. 19-34-17², J. Levy, L. J. 30, 13, p. Barium
Warren, C. I. 32, #0772. Add 10 ml. HNO₃ (d. 1.4)
over 45 min. to 25.5 g. 1-C₆H₅Ph (I), stirring 0.5 hr.
and diln. with H₂O gave 0.5% products, b.p. 160-70°, which
yielded 49% mainly 4-nitro-deriv. (II), b.p. 161-3°. To 6
ml. HNO₃ (d. 1.4) was added at -40° in 10 min., 2.5 g. I,
after which the mixt. was quenched in H₂O yielding after
extn. with Et₂O and washing with Na₂CO₃, 3.8 g. oil, which
chromatographed on Al₂O₃ and eluted successively with petr.
ether, C₆H₆ and EtOH gave in order: 79.8% II, 6.6%
1,4-dinitro-deriv. (III), m. 171-2° (from EtOH), and 11.3%
Oxidation of II with 11% HNO₃ 8 hrs. at 165-73° gave 1,
3-dinitro-1-naphthoic acid, m. 222-3°. To 2 ml. HNO₃ (d.
1.48) was added in 3 min. 0.8 g. I at -10°; after 10 min.
the mixt. was dried, and worked up chromatographically as
above yielding 4.6% II and 71.3% III, m. 171-2°, identical
to the above described; the latter oxidized as above with
11% HNO₃ at 170° gave 1,3-dinitro-1-naphthoic acid,
m. 258°. To 2 ml. HNO₃ (d. 1.5) was added in 10 min. at
-10° 0.5 g. I and after 10 min. stirring at -10° the mixt.
was dried, and worked up as above yielding 50% II and 23%
1,3-dinitro-deriv., m. 195-200° (from C₆H₆).
The material, m. 126-0°. To 2 ml. HNO₃ (d. 1.48)
added at -10° 1 g. II and after stirring 10 min. the mixt.
gave after the usual treatment 20% II and 60% III. To
2 ml. HNO₃ (d. 1.5) was added in 5 min. at 20° 1 g. II and
after stirring 10 min. the mixt. yielded 65.6% 1-trimesic
deriv., m. 198-9°. To 2 ml. HNO₃ (d. 1.5) was added at
-10° 1 g. III and after 10 min. the mixt. was worked up
yielding 58.4% III and much tar. Hydrogenation of II

Sergievskaia, S.I., S. F. Novototskaya, G.Ya.

In EtOAc and EtOH over Raney Ni at 25-40° at atm. pressure gave on distn. in presence of Et 2-mercapto-4-amino-benzoate inhibitor, 80% 4-amino-1-ethylnaphthalene, b.p. 130-1°; δ deriv. m. 149-50°; Et deriv. m. 201-4°.

Heating the amine with Me₂NPh and ClCO₂Et gave Et 1-ethyl-4-naphthalene-carbamate, m. 84-8° (from petr. ether).

G. M. Kosolapoff

Distr: 4E4j/4E3d

✓Chloromethylation of 1-alkynaphthalenes and some transformations of 4-chloromethyl-1-alkynaphthalenes. S. I. Sergievskaya and T. S. Safonova (S. Ordzhonikidze All-Union Chern. Physic. Sci. Research Inst., Moscow). Zhur. Obshchey Khim. 27, 1645-50 (1957). Heating 25 g. 1-CuH₂Bu and 17 g. paraformaldehyde with 6 g. ZnCl and 60 ml. concd. HCl at 65-70° in a stream of dry HCl gave after several distns, 81% 4-chloromethyl-1-butynaphthalene (I), b₁ 140-1°. Similarly were prep'd. 4-chloromethyl-1-ethynaphthalene, b₁ 110-20°; 1-Pr analog, b₁ 140-1°, and 1-iso-Pr analog, b₁ 126-7°. These heated with Pb(OAc)₂ in AcOH 2 hrs. at 90° gave 80-80% 4-acetoxy-1-butynaphthalene, b₁ 140.5-50°; 1-Et analog, b₁ 134-5°; 1-Pr analog, b₁ 150-1°; 1-iso-Pr analog, b₁ 140-50°. These refluxed with alc. KOH 0.5 hr. gave 90-8% 4-hydroxymethyl-1-butynaphthalene, m. 89.5-90°; 1-Et analog, m. 81.5-2°; 1-Pr analog, m. 88.5-9°; 1-iso-Pr analog, m. 84-5°. These oxidized with KMnO₄ in refluxing Me₂CO gave 90-5% 1-butynaphthalene-4-carboxylic acid, m. 148-8.5°; 1-Et analog, m. 129-30°; 1-Pr analog, m. 141-2°; 1-iso-Pr analog, m. 153-3.5°. Heating 15 g. I with 6.2 g. HN(CH₂CH₂OH), 6.5 hrs. at 90°, cooling, treating with 30 g. SOCl₂ in C₆H₆, and heating 1.5 hrs. at 68-70° gave after treatment with dry HCl in Et₂O 10.6 g. N-1-butyl-4-naphthylmethyl-N,N-bis(2-chloroethyl)amine HCl salt (II), m. 129.5-30.5°; the free base was an undistillable oil; similarly were prep'd. analogs of II: 1-Et, m. 147-8°; 1-Pr, m. 158.5-9°; and 1-iso-Pr, m. 120.5-30.5°. G. M. Kosolapoff

SOV/ 79-28-6-37/63

AUTHORS: Sventsitskaya, L. Ye., Kropacheva, A. A., Sergiyevskaya, S.I.

TITLE: 2,4-Di-(Ethylenimino)-1,3,5-Triazine (2,4-di-(etilenimino)-
-1,3,5-triaziny)

PERIODICAL: Zhurnal obshchey khimii, 1958, Vol. 28, Nr 6, pp. 1601-1607
(USSR)

ABSTRACT: Among the 1,3,5-triazine products the 2,4,6-triethylenimino-
-1,3,5-triazine has been well known for a long time as me-
dicament against some kinds of carcinoma; this caused the
upshot of a great number of analogous compounds and supplied
a great contribution of new data to the chemistry of ethylen-
imino triazines. In their search for better remedies against
carcinoma the authors synthesized the 2,4-di-(ethylenimino)-
-1,3,5-triazines which in the third substituent (R) in the
cycle of triazine either contain a nitrogen-containing hetero-
cyclic radical or as radical the ester of an aliphatic or
aliphatic-aromatic amino acid (see scheme 1). In the synthe-
sis of the substituted 1,3,5-triazines cyanuric chloride usual-
ly serves as initial material, in which the chlorine atoms

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2,4-Di-(Ethylenimino)-1,3,5-Triazine

SOV/79-28-6-37/63

are either completely or only partially substituted by other groups or radicals. The substitution can be carried out in different ways in dependence on the character of the reacting compounds and on the conditions of reaction (Ref 1). Two ways could be taken to synthesize the compounds chosen: 1) To synthesize the already known 2,4-di-(ethylenimino)-6-chloro-1,3,5-triazine and to substitute the chlorine by corresponding amino acids in it; or 2) First to substitute only one chlorine atom in the cyanuric chloride by the amino compound in order to then react it on the substituted 2,4-dichlorotriazine with ethylenimine (see scheme 2). Both methods were used. Thus the 6-substituted 2,4-di-(ethylenimino)-1,3,5-triazines were obtained. Those substituted were nitrogen-containing heterocycles and esters of aliphatic and aliphatic-aromatic amino acids. In general the biologic properties of these products are similar to those of triethyleniminotriazine without having special medical-clinical advantages as compared to those already used in medical practice. There are 3 references, 0 of which are Soviet.

Card 2/3

2,4-Di-(Ethylenimino)-1,3,5-Triazine

SOV/79-28-6-37/63

ASSOCIATION: Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy institut im. S. Ordzhonikidze
(All-Union Scientific Chemo-Pharmaceutical Research Institute imeni S. Ordzhonikidze)

SUBMITTED: May 19, 1957

1. Triazines--Synthesis

Card 3/3

AUTHORS: Sergiyevskaya, S. I., Levshina, K. V., Chizhov, A. K., Gavrilova, A. I., Kravchenko, A. I. SOV/79-28-7-24/64

TITLE: N-Di(Ethyl Chloride) Amines of the Alicyclic Series. I(N-Di(khloretil) aminy alitsiklicheskogo ryada. I)

PERIODICAL: Zhurnal obshchey khimii, Vol 28, Nr 7, pp. 1839 - 1845 (USSR)

ABSTRACT: The authors discuss the synthesis and some properties of the dichloroalkylamines of the cyclopentane-, cyclohexane- and cycloheptane series. They synthetized the compounds of two types: In the one (Formula I) the di(chloroalkyl) amino group is directly bound to the carbon of the nucleus, and in the other to the carbon of the side chain (II). The compounds of type (II) are alicyclic derivatives of methyl-N-bis (ethyl chloride) amine which is of importance for medicine. The two methods used most were employed for the synthesis of N-di(ethyl chloride) amine: according to the one [= (a) of Table 1] ethylene oxide reacts with the amino compounds; according to the other [= (b) of Table 1] the compounds containing halogens are caused to react with diethanol amine. The final stage, i.e. the substitution of the hydroxyl groups by chlorine is the same

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N-Di(Ethyl Chloride) Amines of the Alicyclic Series. I SOV/79-28-7-24/64

for both methods, according to the specific characteristic features of the N-di(oxyethyl)amines. The synthesis of the dichloro-alkyl amines of type (I) had to be carried out according to method (a). The necessary alicyclic amines as initial products had been obtained in the cyclopentane- and cycloheptane series by the reduction of the ketone oximes, and in the cyclohexane series by the catalytic hydration of the aromatic amino compounds. The chloro-methyl derivatives of the same alicyclic hydrocarbons served as initial products for the synthesis of the compounds of type (II). The chloro-methyl cycloalkanes were obtained according to the reaction scheme mentioned. Thionyl chloride served as chlorination agent (I and II)(substitution of hydroxyl by chlorine).There are 2 tables and 8 references, 2 of which are Soviet.

ASSOCIATION: Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy institut imeni S.Ordzhonikidze (All-Union Scientific Chemical and Pharmaceutical Institute imeni S. Ordzhonikidze)

Card 2/3

N-Di(Ethyl Chloride) Amines of the Alicyclic Series. I SOV/79-28-7-24/64

SUBMITTED: February 7, 1957

1. Dichloroalkylamines--Synthesis 2. Dichloroalkylamines--Properties
3. Cyclic compounds--Molecular structure 4. Ethyl chloride amines
--Chemical properties

Card 3/3

7/7/84-7-27/14

AUTHORS: Sergiyevskaya, N. I., Lavshina, L. V., Gavrilova, N. I.,
Chizhev, A. I.

TITLE: N-Di (Chloro-Ethyl) amines with Aicyclic and Aromatic Radicals in the Molecules. I: (N-di(Chloro-ethyl)amino's alitsiklicheskimi i aromatichimi radikalami v molekulakh. II)

PHYSICAL: Zhurnal obshchey khimii, 1958, Vol. 28, No 7, pp. 1845-1849
(USSR)

ABSTRACT: The aim of the present investigation was the synthesis of the N-di(chloro-ethyl)amines which simultaneously have an aromatic and an aicyclic radical in the molecule. The structures of these compounds may be seen from the reaction scheme: the compounds (I) and (II) appear as arylated analogs of some N-di(enole-ethyl)amines of the aicyclic series already earlier described by the authors (Ref 1). The compounds (III) differ from (I) and (II) by the fact that the aromatic radical is not a component of the aicyclic radical. The corresponding cyrogen compound serves as initial product, viz., the nitriles AR-CN for the types (I) and (II), and the nitrile

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30.VII.1987-7-16, ed
N,N'-bis-(*p*-chloroethyl) amines with diisocyanates and aromatic Radicals in the
macrocycles. II

R-CH-CN, for type (III), where R denotes an alicyclic radical.



All these nitriles are easily obtained by the condensation of the cyanobenzene with 1,4-dibromobutane, 1,3-dibromopentane and bromocyclohexane in the presence of sodium amide. The reduction of the nitriles to primary amines was carried out either catalytically with hydrogen or by means of lithium-aluminum hydride. The transition from amines to their N-di(ethyloxy)-derivatives and from these to the N-di(chloroethyl)-amines took place according to reference 1. In the purification of the hydrogen chloride salts of the above mentioned amines the solvents had to be selected carefully. The authors synthesized the hitherto not described N-di(chloroethyl)-amines and some other compounds of the cyclopentane- and cyclohexane series. There are 1 table and 5 references, 3 of which are Soviet.

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SOV/79-28-7-25/64
N-Di (Chloro-Ethyl) Amines With Alicyclic and Aromatic Radicals in the
molecules. II

ASSOCIATION: Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmaceuti-
cheskiy institut imeni S. Ordzhonikidze (All-Union Scientific
Chemical and Pharmaceutical Research Institute imeni
S. Ordzhonikidze)

SUBMITTED: February 7, 1957

1. Ethyl chloride amines--Molecular structure 2. Ethyl chloride
--Synthesis 3. Cyclic compounds--Chemical properties

Card 3/3

SHCHUKINA, M.N., prof.; MASHKOVSKIY, M.D., prof.; PERSHIN, G.N., prof., laureat Stalinskoy premii, otv.red.; SERGIYEVSKAYA, S.I., prof., red.; MAGIDSON, O.Yu., prof., laureat Stalinskoy premii, red.; UTKIN, L.M., prof., red.; GROZDEVA, Ye.I., red.; LYUDKOVSKAYA, N.I., tekhn.red.

[Chemistry and medicine] Khimiia i meditsina. Otv.red. G.N. Pershin. Moskva, Medgiz. No.9. [Aminazine] Aminazin. 1959. (MIRA 12:6) 241 p.

1. Moscow. Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy institut. 2. Zaveduyushchaya laboratoriya protivotuberkuleznykh soyedineniy Vsesoyuznogo nauchno-issledovatel'skogo khimiko-farmatsevticheskogo instituta imeni S.Ordzhonikidze (for Shchukina). 3. Zaveduyushchiy laboratoriya otdela farmakologii Vsesoyuznogo nauchno-issledovatel'skogo khimiko-farmatsevticheskogo instituta imeni S.Ordzhonikidze (for Mashkovskiy).

(CHLORPROMAZINE)

CHERNOV, V.A.; LYTKINA, V.B.; SERGIYEVSKAYA, S.I.; KROPACHEVA, A.A.;
PARSHINA, V.A.; SVENTSITSKAYA, L.Ye.

On the antitumor activity of certain derivatives of the trimer and
tetramer of phosphonitrile. Farm. i toks. 22 no.4:365-367 Jl-AB '59.
(MIRA 13:1)

1. Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy
institut imeni S. Ordzhonikidze.

(HETEROCLIC COMPOUNDS pharmacol.)
(ANTINEOPLASTIC AGENTS pharmacol.)

5(3)
AUTHORS:Levshina, K. V., Chizhov, A. K.,
Sheynker, Yu. N., Sergiyevskaya, S. I.

SOV/79-29-4-31/77

TITLE:

Sulfonic Esters of the Cyclohexane Diols and the 1,4-Butane
Diol (Sul'fonovyye estiry tsiklogeksandiola)

PERIODICAL:

Zhurnal obshchey khimii, 1959, Vol 29, Nr 4, pp 1184-1188
(USSR)

ABSTRACT:

Some alkyl sulfonic esters of aliphatic diols proved to be useful active agents against some kinds of cancer. The authors had to decide whether the amount and structure of the radical of sulphur had any effect on the biological properties of the sulfonic esters of 1,4-butane diol, and whether the diol necessarily belonged to the aliphatic series. Alkyl sulfonic esters of 1,4-butane diol with the radicals C_2H_5 , C_3H_7 , cyclo- C_6H_{11} and alkyl sulfonic esters of the isomeric cyclohexane diols (1,2;1,3;1,4) were synthesized. All these compounds were obtained through a transformation of the corresponding sulfochlorides with the diols in water-free benzene and in the presence of triethyl amine. The synthesis of the sulfochlorides was carried out according to references 2 and 3. The initial

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Sulfonic Esters of the Cyclohexane Diols and
the 1,4-Butane Diol

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the synthesized 1,4-alkyl sulfonates of the cyclohexane (methyl-, ethyl, propyl sulfonates) does not cause any sizable changes in the spectrum (Fig 3), it may be assumed that various alkyl sulfonates exhibit the very same configuration, and that the form in question is the stable trans-form. The biological properties of the compounds obtained generally correspond to those of "milerane" (Mileran). There are 3 figures, 1 table, and 8 references, 3 of which are Soviet.

ASSOCIATION: Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy institut imeni S. Ordzhonikidze. (All-Union Scientific Chemopharmaceutical Research Institute imeni S. Ordzhonikidze)

SUBMITTED: February 10, 1958

Card 3/3

SAFONOVA, T.S.; SERGIYEVSKAYA, S.I.

Aryl amides of aliphatic amino acids. Part 2: Aryl amides of
glycine and glycylglycine. Zhur. ob. khim. 30 no.6:1848-1855
Je '60. (MIRA 13:6)

1. Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy
institut imeni S. Ordzhonikidze.
(Amides) (Glycine)

SAFONOVA, T.S.; SERGIYEVSKAYA, S.I.

N-di(chloroethyl) amides of aminocarboxylic acids and their peptides. Part 1: Method of obtaining the N-di(chloroethyl) amide of glycine and of compounds related to it. Zhur.ob. khim. 30 no.7:2432-2433 Jl '60. (MIRA 13:7)

1. Vsesoyuznyy nauchno-issledovatel'skiy khimikofarmatsevти-
cheskiy institut imeni S. Ordzhonikidze.
(Amides) (Glycine)

KROPACHEVA, A.A.; FARSHINA, V.A.; SERGIYEVSKAYA, S.I.

Derivatives of ethylenimine. Part 2: Ethylenimides of phosphoric
and thiophosphoric acids. Zhur. ob. khim. 30 no.11:3584-3588 N'60.
(MIRA 13:11)

1. Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy
institut imeni S.Ordzhonikidze.
(Ethylenimine) (Phosphoric acid) (Phosphorothioic acid)

LEVSHINA, K.V.; GAVRILOVA, A.I.; SERGIYEVSKAYA, S.I.

Bis (β -chloroethyl) amines of bicyclic compounds. Part 1:
Bis (β -chloroethyl) amines of the indan series. Zhur. ob.
khim. 30 no.11:3634-3639 N'60. (MIRA 13:11)

1. Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy
institut imeni S.Ordzhonikidze.
(Amines) (Indan)

CHIZHOV, A.K.; LEVSHINA, K.V.; SERGIYEVSKAYA, S.I.

Bis (β -chloroethyl) aminomethyl derivatives of azobenzene. Part
1: Method of synthesizing bis (β -chloroethyl) amines of 4-substituted-
4'-methylazobenzene. Zhur. ob. khim. 30 no.11:3695-3700 N'60.
(MIRA 13:11)

1. Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskii
institut imeni S. Ordzhonikidze.
(Azobenzene)

CHIZHOV, A.K.; LEVSHINA, K.V.; SERGIYEVSKAYA, S.I.

Bis (β -chloroethyl) amines of bicyclic compounds. Part 3:
Some derivatives of benzocycloheptane with substituents in
position 7 of the bicyclic compound. Zhur. ob. khim. 30 no.11:3700-3702
(MIRA 13:11)
N'60.

1. Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy
institut imeni S. Ordzhonikidze.
(Cycloheptabenzen)

SAFONOVА, T.S.; SERGIYEVSKAYA, S.I.

N, N-bis(β-chloroethyl)amides of aminocarboxylic acids. Part 1:
N, N-bis(β-chloroethyl)glycinamide and compounds related to it.
Zhur. ob. khim. 31 no.4:1193-1199 Ap '61. (MIRA 14:4)

1. Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy
institut imeni S. Ordzhonikidze.
(Glycinamide)

CHIZHOV, A.K.; LEVSHINA, K.V.; SERGIYEVSKAYA, S.I.

Bis (β -chloroethyl) aminomethylazobenzenes and some analogous
compounds. Zhur. ob. khim. 31 no.4:1288-1297 Ap '61.

(MIRA 14:4)

1. Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy
institut imeni S. Ordzhonikidze.
(Azobenzene)

LEVSHINA, K.V.; KOLODKINA, I.I.; SERGIYEVSKAYA, S.I.

N-bis(chloroethyl)amines of bicyclic compounds. Part 5:
Some new derivatives of indan, tetrahydronaphthalene, and
benzocycloheptane. Zhur.ob.khim. 32 no.2:464-467 F '62.
(MIRA 15:2)

1. Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsev-
ticheskiy institut imeni S.Ordzhonikidze.

(Indan)

(Naphthalene)

(Benzocycloheptane)

KROPACHEVA, A.A.; SAZONOV, N.V.; SERGIYEVSKAYA, S.I.

Derivatives of ethylenimine. Part 4: Diethylenimides
of pyrimidine-2-aminophosphoric acids. Zhur. ob. khim.
32 no.11:3796-3799 N '62. (MIRA 15:11)

1. Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy
institut imeni S. Ordzhonikidze.
(Pyrimidine) (Phosphoric acid) (Aziridine)

KOLODKINA, I.I.; LEVSHINA, K.V.; SERGIYEVSKAYA, S.I.

Bis (β -chloroethyl) amines of bicyclic compounds. Part 6:
4- and 5-N-bis (β -chloroethyl) aminoindans. Zhur. ob. khim.
(MIRA 16:2)
33 no.2:469-474 F '63.

1. Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy
institut imeni S. Ordzhonikidze.
(Indian) (Cyclic compounds) (Amines)

SAPONOVA, T.S.; SERGIYEVSKAYA, S.I.

Arylamides of aliphatic amino acids. Part 3:p-N-di(chlcroethyl)
aminophenylamides of some amino acids. Zhur. ob. khim. 34 no. 3:
919-923 Mr '64. (MIRA 17:6)

1. Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy
institut imeni S.Ordzhonikidze.

CHIZHOV, A.K.; LEVSHINA, K.V.; SERGIYEVAYA, S.I.

Bis(β -chloroethyl) aminomethyl derivatives of azobenzene.
Part 3: p-Hydroxy-m-(o)-carboxy-p'-bis(β -chloroethyl)-
aminomethylazobenzenes, their derivatives and analog compounds.
Zhur. ob. khim. 34 no. 5:1587-1592. Ny '64. (MIRA 17:7)

1. Vsesoyuznyj nauchno-issledovatel'skiy khimiko-farmatsevticheskij
institut imeni S.Ordzhonikidze.

X

SAFONOVA, T.S.; SIRGIYEVSKAYA, S.I.

N,N'-bis(β -chloroethyl) amides of aminocarboxylic acids. Part 2:
N-substituted amides of N-phthaloyl and N'-tritylalanines. Zhur.
org.khim. 1 no.3:450-454 Kr '65, (MIRA 18:4)

1. Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy
institut im. S.Ordzhonikidze.

POLIKOVSKIY, V.I., doktor tekhnicheskikh nauk; SERGIYEVSKAYA, T.G., inzhener;
AL'PER, T.I., inzhener; MACHEKHINA, G.N., inzhener.

Aerodynamics of the cooling systems of large hydraulic generators.
Vest.elektroprom.27 no.1:9-16 Ja '56. (MLRA 9:6)

1.Nauchno-issledovatel'skiy institut Ministerstva elektropromyshlennosti.
(Electric generators--Cooling) (Fans, Mechanical)

SERGIYEVSKAYA, T. G. Cand Tech Sci -- (diss) " Certain ^{problems} questions
in aerodynamics of systems of cooling electric machines." Mos, 1957.
20 pp with diagrams 22 cm. (Min of Higher education. Mos Order of
Lenin Power Engineering Inst im V,M. Molotov). (KL, 23-57, 113)

-85-

80

AUTHOR: Sergiyevskaya, T.G., Engineer

TITLE: Cooling the Stator of a Large Hydrogenerator
(Ob okhlazhdenii statora krupnogo gidrogeneratora)

PERIODICAL: Vestnik Elektropromyshlennosti 1957, No.2, pp.38-43
(U.S.S.R.)

ABSTRACT: Efficient ventilation is a difficult problem in large hydro-alternators but methods of achieving great improvements are now becoming available. The main problem of air flow distribution occurs in the stator. In some machines the cooling of stator windings is not uniform along the entire length of the slot and local hot spots occur. Non-uniform air flow is sometimes observed in stator ducts and the rate of air flow from top and bottom is often different. This article presents briefly the results of an investigation of the distribution of the flow of coolant in the stator ducts of a large generator. The objective was to obtain a qualitative physical analysis of the processes

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TITLE: Cooling the Stator of a Large Hydrogenerator
(Ob okhlazhdenii statora krupnogo gidrogeneratora)
which govern this distribution and to develop an
approximate method of computation.

The process was investigated experimentally on special models and rigs proceeding from simple to more complicated cases. Factors affecting the rate of air flow were studied, and methods of air supply which gave more uniform distribution were found. It was found that increasing the fan output to increase the flow of air could increase the non-uniformity of cooling and cause reduction in the flow through the rotor ducts. The reasons for assymetrical flow in certain generators was investigated. Methods of reducing the aerodynamic resistance of the stator were found.

The process of air flow in the stator ducts can be computed approximately; the necessary assumptions are made and a differential equation is formulated.

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TITLE: Cooling the Stator of a Large Hydrogenerator
(Ob okhlazhdenii statora krupnogo gidrogeneratora)

A method of calculating the distribution of air is
then worked out.

The method was used to compute air flow in a model of
a hydro-alternator and also in one of the generators
at the Dnepr Hydroelectric Station with air supplied
from the two ends of the machine to the air gap and
to the space between the poles. The results of air
velocity in the ducts along the stator are plotted in
the form of a graph, and there is reasonable agree-
ment between theory and experiment.

Reference, in a footnote is made to the following:
T.I. Al'per, G.N. Machekhina and A.A. Minaev.

The article contains 6 diagrams, 1 photograph and
1 graph.

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TITLE: Cooling the Stator of a Large Hydrogenerator
(Ob okhlazhdenii statora krupnogo gidrogeneratora)

ASSOCIATION: Scientific Research Institute of the Ministry of
Electrotechnical Industry **НИИ МЭП** (Naucho-Issledo-
vatel'skiy Institut Ministerstva Elektrotekhnicheskoy
Promyshlennosti)

PRESENTED BY:

SUBMITTED:

AVAILABLE: Library of Congress

Card 4/4

110-10-7/18

AUTHOR: Polikovskiy, V.I., Doctor of Technical Sciences, Professor,
and Sergiyevskaya, T.G., Al'per, T.I., Engineers.

TITLE: An investigation of systems of cooling a hydro-alternator
using a ventilation model. (Issledovaniya sistemy okhla-
zhdeniya gidrogeneratora na ventilyatsionnoy modeli.)

PERIODICAL: Vestnik Elektropromyshlennosti, 1957, Vol.28, No.10,
pp. 35 - 44 (USSR)

ABSTRACT: The problem of modelling the cooling system of a large
hydro-alternator arose in connection with the design of mach-
ines for the Kuybyshev and Stalingrad Power Stations which were
of considerably greater output than the largest generators
previously built. It is difficult to make investigations on
existing hydro-alternators because the important parts are
inaccessible and it is not possible to change the operating
conditions of the cooling system or to alter the design of
the systems. On models these limitations are easily overcome.
Complete modelling of a machine is a complicated task but it
is much simpler to model only the cooling. In order to model
electro-thermal processes it is generally necessary to model electro-
magnetic processes. However, the method of calculation of
thermal losses of electro-magnetic origin is sufficiently
accurate and therefore modelling of electro-magnetic processes
can be avoided. When considering the problem to a first
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An Investigation of Systems of Cooling a Hydro-alternator Using a Ventilation Model.

approximation, it is also possible to avoid modelling the thermal processes in the system since, with the relatively small change of temperature of the air in the machine, the thermal processes have little influence on the hydraulic processes and the influence of the speed and distribution of air flows in the ducts of the system on the heat transmission can be taken into account approximately by modelling individual components of the system with subsequent thermal calculations. Thus, the task is reduced to hydraulic modelling of the complete cooling system of a hydro-alternator and its parts, thermal modelling of individual components of the machine and calculations. This article describes the first stage of a complex hydraulic investigation on the cooling system of a hydro-alternator using a ventilation model. The work was carried out in the Scientific Research Institute of the Electro-technical Industry under the leadership of Prof. V.I. Polikovskiy, with the participation of Engineers S.I. Lyubintsev and G.N. Machekhina, as well as the authors of the article.

It was decided to model a hydro-alternator of the type used in the Dneproges Station because the results obtained on the Card 2/9 model could be compared with those of ventilation tests on the

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An investigation of Systems of Cooling a Hydro-alternator Using a Ventilation Model.

actual generator. The complete cooling system of the generator was modelled on the assumption that auto-modelling of the system was possible, that is that the resistance coefficient is independent of the Reynolds number. Investigations on the model fully confirm the validity of this assumption: for all tests on the model Euler's parameter remained independent of the speed of rotation of the model. In auto-modelling systems, to ensure physical similarity between the hydraulic processes in the model and in the actual machine it is sufficient that they should be geometrically and kinematically similar. The results of measurements of pressure and rate of air flow on the model recalculated to full scale are in good agreement with the results of tests on the actual generators. The results are compared in Table 1.

The model investigated is a ventilation model of a large generator made to a scale of one-fifth, geometrical similarity being maintained in the main parts. A picture of the model is given in Fig. 1, a diagram in Fig. 2 and illustrations of the stator and rotor in Figs. 3 and 4. The model was driven by a wound rotor induction motor of 55 kW. The model is described Card 3/9 in detail; the arrangement of the fan blades is shown in Fig. 5.

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An Investigation of Systems of Cooling a Hydro-alternator Using a Ventilation Model.

The model was investigated with open-cycle air-circuit which considerably simplified the test procedure without much affecting the results. The main object of the hydraulic investigation of the cooling system on the model was to obtain a physical picture of the operation of the system, particularly: to find out the distribution of air flow and pressure in the system ducts; to determine characteristics of the main head-producing parts of the system such as the fan and rotor and to investigate their inter-relationship; and to determine the stator characteristics.

Detailed consideration is then given to the operation of the fans which consist of centrifugal wheels with straight radial blades as shown in Fig. 4 such as are installed on hydro-alternators of the Dneproges and other stations. The experimental characteristics of the centrifugal fans on the model are shown in Fig. 6. During the tests on a fan, the other fan and the rotor were put out of operation by appropriately blocking the air inlets. The results show that the total theoretical head of the fans under design conditions of operation is about 220 mm of water, the dynamic head at the fan outlet 125 mm of water, Card 4/9 the loss of head is 75 mm of water and the useful static head

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An Investigation of Systems of Cooling a Hydro-alternator Using a
ventilation Model.

of the fan is 20 mm of water. Most of the hydraulic losses consist of loss at the inlet of flow to the wheel. They result from the large angles of attack of the blades and the large inlet diameters. The curves in Fig. 7a show the relationship between the relative rate of flow at the inlet to the wheel and the inlet diameter at a given flow. It is shown that to increase the useful static head of the fan the blades should be bent round at inlet and the inlet diameter of the wheel should be reduced to the optimum value. The investigations on the model show that the static pressure beyond the centrifugal wheel continues to increase in the end winding chamber as shown in Fig. 8. This part of the system is then working as a guide vane apparatus in which the dynamic head of the rotating flow is partially converted into a static head. The greater the static pressure in the end winding chambers the greater the flow delivered to the inter-pole space of the model from the ends of the poles. Experimental characteristics of the rotor pole are shown in Fig. 10 which demonstrate the use of guide pieces to direct the air flow.

The operation of the rotor as a fan is then considered. The Card 5/9 rotor may be considered as a combination of fans operating in

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An Investigation of Systems of Cooling a Hydro-alternator Using a ventilation Model.

series and parallel. A diagram of the ventilating channels in the rim is shown in Fig. 11 which gives the experimental characteristics of the rotor with mean static head and flow through the screens. The characteristics which are obtained, together with visual observations, make it possible to analyse the operation of the rotor as a whole and of its component parts. At small rates of flow the rotor pressure is high and the quantity of air that flows out through the ends of the inter-pole faces is greater than the quantity flowing in, which corresponds to points to the left of the intersection of the curve on Fig. 11. At high rates of flow the quantity of air flowing out of the ends decreases which corresponds to that part of the rotor characteristic in Fig. 11 to the right of the intersection of the curves. These parasitic circulations cause additional power losses in ventilation and since it is hot air that is circulated they must impair the removal of heat from the machine. The distribution of static pressure in the gap along the model with combined operation of the rim channels and the ends of the pole is shown in Fig. 12. Investigations of pressure distribution in the gap show that it is non-uniform because of Card 6/9 the delivery of air through the stator ducts in the presence of

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An Investigation of Systems of Cooling a Hydro-alternator Using a Ventilation Model.

the change of resistance coefficients of the stator as a function of the ratio of the mean speed of the air in the stator ducts to the speed of rotation of the air in the gap are given in Fig.13. The problem of reducing the resistance of the stator by altering the construction of the ducts is of interest. Curves of the mean velocity distribution at the outlet from the stator ducts are given in Fig.14. The curves show that the flow distribution through the stator ducts is not uniform around the model and moreover it is asymmetrical.

Special tests show that distribution of flow round the stator is determined, to a first approximation, by the distribution of static pressures in the gap and the asymmetry of flow around the stator is associated with differences between the characteristics of the upper and lower fans of the model and also with the asymmetry of the air ducts of the system.

As a result of the investigations it is possible to obtain a physical picture of the operation of the cooling systems of large hydro-alternators. The material obtained can serve as a basis for systematic work on the improvement of existing systems and for the development of design procedures for them.

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The following are the most promising directions of work:

110-4-1/25

AUTHORS: Polikovskiy, V.I., Doctor of Technical Sciences, Professor,
Al'per, T.I., Engineer, Zemlyanoy, M.S., and Sergiyevskaya,
T.G., Candidates of Technical Sciences.

TITLE: A New Method of Cooling Large Hydro-alternators (Novaya skhema
okhlazhdeniya krupnykh gidrogeneratorov)

PERIODICAL: Vestnik Elektropromyshlennosti, 1958, No. 4,
pp. 1 - 5 (USSR).

ABSTRACT: In designing hydro-alternators for 200 - 300 MW, improved
cooling methods became necessary. At present, the fan effect
of the rotor spider is not effectively used, nor are the centri-
fugal fans well designed.

The article describes a new construction in which the spaces
between the arms of the rotor spider are partly enclosed, but
apertures are left near the hub to entrain cooling air. Near
the extremities of the arms, the shrouding stands away in the
form of an inclined flange, leaving a circumferential space.
This is divided by radial vanes and the passages so formed
assist in drawing the cooling air centrifugally outwards and
direct some of it across the ends of the rotor and stator coils.
With this design the air-flow through the hydro-alternator is
about 40% greater than that given by the usual type of fan.

Card 1/3 Performance characteristics of the old and new cooling arrangements are graphed in Fig. 2.

A new Method of Cooling Large Hydro-alternators 110-4-1/25*

The new system is more effective because the lower relative air-speed at the air inlet (to the rotor) reduces the losses, so that the discharge pressure is greater. The effectiveness of the system depends on the position of the intake aperture; the position is chosen to give the minimum air velocity at the inlet for a given flow. Values are plotted in Fig.3. A design procedure for the new type of fan is given, with appropriate formulae. The flow round the ends of the winding is depicted in Fig.5.

variants of the new system were tested. In particular, experiments were made with air entering the generator from only one side. Test results for this case, plotted in Fig.6, show that the performance is about the same as when entry is from both sides. It follows that when the inlet area is of the optimum value it does not matter whether intake is from one side or two. The main defect of existing ventilating systems is the large inlet diameters of the fans, which cause high losses. Other ways of overcoming this difficulty besides the one described are possible and are briefly mentioned.

The new method of ventilation was tried on one of the hydro-alternators of the Gor'kiy Hydroelectric Power Station (Gor'kovskaya GES) and comparative tests confirmed the correctness of the
Card 2/3

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POLIKOVSKIY, V.I., doktor tekhn.nauk, prof.; SERGIYEVSKAYA, T.G.,
kand.tekhn.nauk

Operational features of axial fans in the cooling systems of large
electrical machinery. Vest. elektroptom. 32 no.12:26-32 D '61.
(MIRA 14:12)

(Electric machinery..Cooling)
(Fans, Electric)

APPROVED FOR RELEASE: 08/23/2000

CIA-RDP86-00513R001548120016-3"

SERGIYEVSKAYA, T.G., kand.tekhn.nauk

Heat emission of the stator of an electrical machine. Vest.
elektroprom. 33 no.11:4-14 N '62. (MIRA 15:11)
(Electric machinery--Cooling)

SELEGINSKAYA, T.G., kand. tekhn. nauk

Heat emission of the frontal connections of the stator winding
of a hydrogenerator. Elektrotehnika 36 no.5:12-14 My '65.
(MIRA 18:5)

POGODIN-ALEKSEYEV, G.I., doktor tekhn.nauk, prof.; SERGIYIVSKAYA, T.V. [deceased]
kand.tekhn.nauk

Effect of microstructure on the development of reversible temper
brittleness in low-carbon manganese steel. Trudy Sek.metalloved.i
term.obr.met.NTO mash.prom. no.2:59-68 '60. (MIRA 14:4)
(Manganese steel--Metallography) (Tempering)

137-58-5-8879

Translation from: Referativnyy zhurnal, Metallurgiya, 1958, Nr 5, p 19 (USSR)

AUTHORS: Sergiyevskaya, Ye. M., Vol'skiy, A. N.

TITLE: To the Theory of Leaching Zinc out of Roasted Zinc Concentrates (K teorii vyshchelachivaniya tsinka iz obozhzhennykh tsinkovykh kontsentratov)

PERIODICAL: Sb. nauchn. tr. Mosk. in-t tsvetn. met. i zolota i VNITO tsvetn. metallurgii, 1957, Nr 26, pp 265-278

ABSTRACT: The dynamic method was employed to study the rate of dissolution of ZnO in H₂SO₄ solutions. The rate of dissolution of ZnO is determined by the diffusion rate when the concentration of H₂SO₄ exceeds 0.36 mole/liter, and by the rate of the chemical reaction itself when the acidity is less. The rate of dissolution decreases if the concentration of ZnSO₄ in the original solution is increased. The rate of dissolution is given as a mathematical function of rate of motion of the sulfuric acid solution. It is shown that at an H₂SO₄ concentration of 0.72 mole/liter and at temperatures between 20°C and 58°C the nature of the process is typically diffusional, the constant of the reaction rate being a linear function of temperature and the temperature coefficient being equal to 1.3.

L. P.

Card 1/1
1. Zinc ores--Processing 2. Zinc--Separation

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Translation from: Referativnyy zhurnal, Metallurgiya, 1958, Nr 8, p 69 (USSR)

AUTHORS: Sergiyevskaya, Ye.M., Vol'skiy, A.N.

TITLE: A Contribution to the Theory of the Leaching of Zinc From Burnt Zinc Concentrates. Kinetics of Dissolution of Copper Oxide in Sulfuric-acid Solutions (K teorii vyshchelachivaniya tsinka iz obozhzhennykh tsinkovykh kontsentratov. Kinetika rastvorenija okisi medi v rastvorakh sernoj kisloty)

PERIODICAL: Sb. nauchn. tr. Mosk. in-t tsvetn. met. i zolota, 1957, Nr 27, pp 102-118

ABSTRACT: A dynamic method is used to study the influence of temperature and H_2SO_4 and $ZnSO_4$ concentrations upon the dissolution rate (DR) of CuO in H_2SO_4 . It is established that the dependence of the DR of CuO upon the concentration of H_2SO_4 in the solution takes on the character of a process of adsorption and is subject to Langmuir's equation for adsorption:
 $v_{1\ hr} = 5.65 [H^+] / (1 + 2.26 [H^+])$. The temperature has a significant influence upon the DR of CuO in H_2SO_4 . The temperature coefficient of the DR is 1.83-1.51. The DR of CuO in H_2SO_4 of

Card 1/2

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A Contribution to the Theory of the Leaching of Zinc (Cont.)

elevated $ZnSO_4$ contents in the initial solution diminishes approximately in proportion to the increase in the $ZnSO_4$ contents of the solution. The energy of activation of the reaction of dissolution of CuO in H_2SO_4 is 10,260 ± 257 cal/mole. The DR of CuO is monitored by the rate of adsorption of H^+ ions or molecules of water onto the surface of the CuO from the solution.

G.S.

1. Zinc--Processing
2. Copper oxide--Chemical reactions
3. Sulfuric acid--Chemical reactions

Card 2/2